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FOREWORD

The *Proceedings* of the American Society for Testing Materials are published annually and include all reports and papers offered to the Society during the current year and accepted by the Administrative Committee on Papers and Publications for the *Proceedings*, together with discussion.

The table of contents and subject and author indexes cover all papers and reports published by the Society during the current year, which in addition to those appearing in the *Proceedings* includes those accepted for publication in the ASTM BULLETIN or as Special Technical Publications.

A list of the Special Technical Publications published by the Society in 1951 is given on the last page of this volume. This supplements the list appearing in the 1948, 1949, and 1950 *Proceedings* covering all special publications published by the Society up to and including 1950.

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SUMMARY OF PROCEEDINGS

OF THE

FIFTY-FOURTH ANNUAL MEETING

ATLANTIC CITY, N. J., JUNE 18–22, 1951

This summary of the Fifty-fourth Annual Meeting of the American Society for Testing Materials, held at the Chalfonte-Haddon Hall Hotel, Atlantic City, N. J., June 18–22, 1951, is a record of the transactions of the meeting, including the actions taken on the various recommendations submitted by the technical committees. In all, 24 technical sessions and 1 informal session were held.

The registered attendance of the meeting is as follows: Members present or represented, 1220; committee members, 660; guests, 402; total, 2282; ladies, 393.

The Proceedings are set forth session by session. There were 60 reports and 109 formal papers presented. The record with respect to each has been briefed, the recommendations in the reports having been grouped so as to cover the acceptance of material for publication as tentative, such as new specifications, methods of test, revisions of tentatives, and proposed revisions of existing standards, and, as a separate group, the approval of matters that were referred to letter ballot of the Society, comprising the adoption of tentatives as standard and the adoption of revisions of standards. Accordingly, wherever the action is reported "adopted as standard" or "adopted as standard, revisions in" it is understood that this indicates approval of the Annual Meeting for reference to letter ballot of the Society. The various recommendations so recorded are included in the Society letter ballot.[1] The actions designated as "accepted as tentative" or "accepted as tentative, revisions in," are self-evident as indicating acceptance by the Society at the Annual Meeting for publication as tentative.

While all the items on the program are recorded under the particular session in which they are presented, for convenience in locating actions with respect to any particular report, the accompanying list is presented of all reports together with the page reference where the actions thereon are recorded:

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[1] The letter ballot on recommendations affecting standards, distributed to the Society membership, was canvassed on September 30, 1951.

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Symposium on Consumer Wants 18
Symposium on Structural Sandwich Constructions 20
Symposium on Acoustical Materials 21

FIRST SESSION

MONDAY, JUNE 18, 2 P.M.

OPENING SESSION

SESSION CHAIRMAN: PRESIDENT L. J. MARKWARDT

Formal Opening of the Fifty-fourth Annual Meeting by President L. J. Markwardt

Approval of Minutes of 1950 Annual Meeting

Report of Board of Directors:

The Executive Secretary made a brief presentation of the Annual Report of the Board of Directors, which had been preprinted. An amendment of the By-Laws was presented for action as given in detail in the Report of the Board of Directors. This amendment proposes that the following sentence in Article I, Section 7, of the By-Laws dealing with honorary members be changed from its present form, namely, "A nominee for honorary membership shall be proposed to the Board of Directors by at least ten members," to read: "Nominees for honorary membership may be proposed in writing to the Board of Directors by any member of the Society." On motion duly seconded the report was accepted for publication in the Proceedings.

Report of Committee E-11 on Quality Control of Materials:

Report presented, in the absence of the chairman, by M. D. Huber and accepted as a report of progress.

SYMPOSIUM ON BULK SAMPLING[2]

SESSION CHAIRMAN: A. E. BRANDT

Introductory Remarks—W. E. Deming.

Materials Handling for Bulk Sampling—Joseph Manuele, presented by the author.

Economic Accumulation of Variance Data in Connection with Bulk Sampling—Louis Tanner and Melvin Lerner, presented by Mr. Tanner.

Two-Stage Acceptance Sampling by Attributes—C. W. Dunnett and J. W. Hopkins, presented from manuscript by Mr. Dunnett.

(*Symposium continued in Third Session*)

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[2] Issued as separate publication *STP No. 114.*

SECOND SESSION—SYMPOSIUM ON CONSOLIDATION TESTING OF SOILS[3]

MONDAY, JUNE 18, 2:30 P.M.

SESSION CHAIRMAN: D. M. BURMISTER

Report of Consolidation Tests with Peat—J. B. Thompson and L. A. Palmer, presented from manuscript by Mr. Palmer.

Consolidation and Related Properties of Loess Soils—W. G. Holtz and H. J. Gibbs, presented from manuscript by Mr. Holtz.

Settlement of a Railroad Embankment Crossing the Morganza Floodway, Louisiana—W. G. Shockley and C. I. Mansur, presented from manuscript by Mr. Mansur.

Aids in the Interpretation of the Consolidation Test—L. H. Matlock, Jr., and R. F. Dawson, presented from manuscript by Mr. Matlock.

A Rapid Technique of Consolidation Testing—R. H. Karol, presented from manuscript by the author.

The Effect of Temperature on the Consolidation Characteristics of Remolded Clay—F. N. Finn, presented from manuscript by R. Horonjeff.

Observed Settlements of Highway Structures Due to Consolidation of Alluvial Clay—E. S. Barber, presented from manuscript by the author.

The Application of Controlled Test Methods in Consolidation Testing—D. M. Burmister, presented from manuscript by the author.

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[3] To be issued as separate publication *STP No. 126*.

THIRD SESSION—SYMPOSIUM ON BULK SAMPLING[2]

(Continued from First Session)

MONDAY, JUNE 18, 8 P.M.

SESSION CHAIRMAN: C. A. BICKING

This session was a continuation of the Symposium on Bulk Sampling. The papers presented at this session are listed below:

Coal Sampling Problems—A. A. Orning, presented by the author.

The Analysis of Variance in a Sampling Experiment—W. M. Bertholf, presented by the author.

The Design of Coal Sampling Procedures—W. M. Bertholf, presented from manuscript by the author.

FOURTH SESSION—FATIGUE AND NON-FERROUS METALS

MONDAY, JUNE 18, 8 P.M.

SESSION CHAIRMAN: G. R. GOHN

Committee B-4 on Electrical Heating, Resistance, and Related Alloys:

Report presented, in the absence of the chairman, by J. S. Pettibone and accepted as a report of progress.

Papers:

The Fatigue Test as Applied to Lead Cable Sheath—G. R. Gohn and W. C. Ellis, presented by Mr. Gohn.

The Influence of Frequency on the Repeated Bending Life of Acid Lead—J. F. Eckel, presented by the author.

Compression Tests on Lead Alloys at Extrusion Temperatures—G. M. Bouton and G. S. Phipps, presented by Mr. Phipps.

The Influence of Cold Work and Heat Treatment on the Engineering Properties of Beryllium Copper Wire—J. T. Richards, R. K. Levan, and E. M. Smith, presented from manuscript by Mr. Richards.

Fatigue Tests in Axial Compression—N. M. Newmark, R. J. Mosborg, W. H. Munse, and R. E. Elling, presented from manuscript by Mr. Mosborg.

Prediction of Relaxation of Metals from Creep Data—Irving Roberts, presented by the author.

FIFTH SESSION—NON-FERROUS METALS

TUESDAY, JUNE 19, 9:30 A.M.

SESSION CHAIRMAN: J. H. FOOTE

Committee B-1 on Wires for Electrical Conductors:

Report presented, by W. R. Hibbard, vice-chairman, and the following actions taken:

Accepted as Tentative:

Spec. for Tinned Hard-Drawn Copper Wire for Electrical Purposes

Accepted as Tentative, Revisions in:

Spec. for Hard-Drawn Copper Wire (B 1 - 49)
Spec. for Rope-Lay-Stranded Copper Conductors Having Bunch-Stranded Members for Electrical Conductors (B 172 - 50 T)
Spec. for Rope-Lay-Stranded Copper Conductors Having Concentric Stranded Members for Electrical Conductors (B 173 - 50 T)
Spec. for Bunch-Stranded Copper Conductors for Electrical Conductors (B 174 - 50 T)

Accepted as Standards, Revisions in:

Spec. for Tinned Soft or Annealed Copper Wire for Electrical Purposes (B 33 - 50)
Spec. for Lead-Coated and Lead-Alloy-Coated Soft Copper Wire for Electrical Purposes (B 189 - 50)
Spec. for Soft Rectangular and Square Bare Copper Wire for Electrical Conductors (B 48 - 49), changing "nominal net areas" to read "working net areas" in NOTES 7 and 8.

Committee B-5 on Copper and Copper Alloys:

Report presented in the absence of the chairman, by G. R. Gohn, and the following actions taken:

Accepted as Tentative:

Spec. for General Requirements for Wrought Copper and Copper Alloy Plate, Sheet, Strip, and Rolled Bar
Spec. for General Requirements for Wrought Copper and Copper Alloy Rod, Bar, and Shape
Spec. for General Requirements for Wrought Copper Alloy Wire
Spec. for General Requirements for Wrought Seamless Copper and Copper Alloy Pipe and Tube

Accepted as Tentative, Revisions in:

Spec. for Cartridge Brass Sheet, Strip, Plate, Bar, and Disks (B 19 - 50 T)
Spec. for Naval Brass Rods, and Shapes (B 21 - 50 T)
Spec. for Brass Plate, Sheet, and Strip (B 36 - 50 T)
Spec. for Seamless Copper Tubes (B 75 - 49 T)
Spec. for Leaded Brass Plate, Sheet, and Strip (B 121 - 50 T)
Spec. for Copper-Nickel-Zinc and Copper-Nickel Alloy Plate, Sheet, and Strip (B 122 - 50 T)
Spec. for Gilding Metal Strip (B 130 - 50 T)
Spec. for Copper Rods, Bars, and Shapes (B 133 - 49 T)
Spec. for Seamless Brass Tubes (B 135 - 49 T)
Spec. for Phosphor Bronze Rods, Bars, and Shapes (B 139 - 50 T)
Spec. for Aluminum Bronze Rods, Bars, and Shapes (B 150 - 49 T)
Spec. for Copper-Nickel-Zinc Alloy Rods and Bars (B 151 - 50 T)
Spec. for Copper Sheet, Strip, and Plate (B 152 - 50 T)
Spec. for Phosphor Bronze Wire (B 159 - 49 T)
Spec. for Aluminum Bronze Sheet and Strip (B 169 - 50 T)
Spec. for Seamless Copper Bus Pipes and Tubes (B 188 - 49 T)
Spec. for Beryllium-Copper Alloy Plate, Sheet and Strip (B 194 - 50 T)
Spec. for Beryllium-Copper Alloy Rod and Bar (B 196 - 49 T)
Spec. for Beryllium-Copper Alloy Wire (B 197 - 50 T)
Spec. for Copper-Nickel-Zinc Alloy Wire (B 206 - 50 T)

Adopted as Standard:

Spec. for Cartridge Case Cups (B 129 - 49 T)
Spec. for Gilding Metal Bullet Jacket Cups (B 131 - 49 T)
Spec. for Copper Bus Bars, Rods, and Shapes (B 187 - 49 T)

Adopted as Standard, Revisions in:

Spec. for Free-Cutting Brass Rod and Bar For Use in Screw Machines (B 16 - 49)
Spec. for Bronze Castings for Turntables and Movable Bridges and for Bearing and Expansion Plates of Fixed Bridges (B 22 - 49)

Spec. for Seamless Copper Pipe, Standard Sizes (B 42 – 49)
Spec. for Seamless Red Brass Pipe, Standard Sizes (B 43 – 49)
Spec. for Steam or Valve Bronze Castings (B 61 – 49)
Spec. for Composition Brass or Ounce Metal Castings (B 62 – 49)
Spec. for Seamless Copper Tubing, Bright Annealed (B 68 – 49)
Spec. for Seamless Copper Water Tube (B 88 – 50)
Spec. for Copper-Silicon Alloy Plate and Sheet for Pressure Vessels (B 96 – 49)
Spec. for Copper-Silicon Alloy Plate, Sheet, and Strip for General Purposes (B 97 – 50)
Spec. for Copper-Silicon Alloy Rods, Bars, and Shapes (B 98 – 49)
Spec. for Copper-Silicon Alloy Wire for General Purposes (B 99 – 49)
Spec. for Phosphor Bronze Plate, Sheet, and Strip (B 103 – 50)
Spec. for Copper and Copper-Alloy Seamless Condenser Tubes and Ferrule Stock (B 111 – 50)
Spec. for Copper and Copper-Base Alloy Forging Rods, Bars, and Shapes (B 124 – 50)
Spec. for Brass Wire (B 134 – 50)
Spec. for Manganese Rods, Bars, and Shapes (B 138 – 49)
Spec. for Leaded Red Brass (Hardware Bronze) Rods, Bars, and Shapes (B 140 – 50)
Method of Mercurous Nitrate Test for Copper and Copper Alloys (B 154 – 50)
Spec. for Copper-Alloy Condenser Tube Plates (B 171 – 49)
Spec. for Locomotive Staybolts (B 12 – 49)
Spec. for Free-Cutting Brass Rod and Bar for Use in Screw Machines (B 16 – 49)
Spec. for Copper-Silicon Alloy Rods, Bars, and Shapes (B 98 – 49)
Spec. for Manganese Bronze Rods, Bars, and Shapes (B 138 – 49)
Spec. for Leaded Red Brass (Hardware Bronze) Rods, Bars, and Shapes (B 140 – 50)

Withdrawal of Tentative:

Spec. for Beryllium-Copper Alloy Strip, Special Grade (B 195 – 50 T)

Committee B-8 on Electrodeposited Metallic Coatings:

Report presented by C. H. Sample, chairman, and the following actions taken:

Accepted as Tentative:

Rec. Practice for the Preparation of Zinc-Base Die Castings for Electroplating
Rec. Practice for the Preparation of and the Electroplating on Aluminum Alloys
Rec. Practice for the Preparation of and Electroplating on Stainless Steel

Accepted as Tentative, Revisions in:

Spec. for Electrodeposited Coatings on Steel (A 164 – 49 T)
Spec. for Electrodeposited Coatings on Cadmium on Steel (A 165 – 49 T)
Methods of Test for Local Thickness of Electrodeposited Coatings (A 219 – 45 T), jointly with Committee A-5 on Corrosion of Iron and Steel

Adopted as Standard:

Spec. for Electrodeposited Coatings of Nickel and Chromium on Zinc and Zinc-Base Alloys (B 142 – 45 T)
Rec. Practice for the Preparation of High-Carbon Steel for Electroplating (B 242 – 49 T)

Committee B-9 on Metal Powders and Metal Powder Products:

Report presented by W. A. Reich, chairman, and the following actions taken:

Accepted as Tentative:

Spec. for Sintered Metal Powder Structural Parts from Bronze

Accepted as Tentative, Revisions in:

Spec. for Metal Powder Sintered Bearings (Oil Impregnated) (B 202 – 45 T)

Papers:

Creep Characteristics of Phosphorized Copper (0.019 per cent P) at 300, 400, and 500 F—A. I. Blank and H. L. Burghoff, presented by Mr. Blank.
Mechanical Properties of Copper at Various Temperatures—W. H. Munse and N. A. Weil, presented from manuscript by Mr. Munse.
Rheotropic Brittleness: General Behaviors—E. J. Ripling and W. M. Baldwin, Jr., presented by Mr. Ripling.
The Scaling of Zirconium in Air—C. A. Phalnikar and W. M. Baldwin, Jr., presented from manuscript by Mr. Baldwin.

SIXTH SESSION—SYMPOSIUM ON FLAME PHOTOMETRY[4]

TUESDAY, JUNE 19, 9:30 A.M.

SESSION CHAIRMAN: L. R. PRITCHARD

A Review of Flame Photometry—V. W. Meloche, presented by the author.

A Stable Internal Standard Flame Photometer for Sodium, Potassium, Lithium, and Calcium Analyses in Biological Fluids and a Study of Ion Interference—C. L. Fox, Jr. and Elizabeth B. Freeman, presented from manuscript by Mr. Fox.

Use of the Beckman and Perkin-Elmer Flame Photometers for the Determination of Alkalies in Portland Cement—J. J. Diamond and Leonard Bean, presented by Mr. Diamond.

Applications of Flame Photometry for the Analysis of Alkalies in Silicates, Waters, and Metals—J. L. Gilliland, presented by R. C. Mielenz.

Determination of Lithium Oxide in Portland Cement by Flame Photometer—W. J. McCoy and G. G. Christiansen, presented from manuscript by Mr. McCoy.

(*Symposium continued in Ninth Session*)

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[4] Issued as separate publication *STP No. 116.*

SEVENTH SESSION—STATISTICAL ASPECTS OF FATIGUE[5]

TUESDAY, JUNE 19, 9:30 A.M.

SESSION CHAIRMAN: R. E. PETERSON

Planning and Interpretation of Fatigue Tests—A. M. Freudenthal, presented by the author.

On the Statistical Nature of Fatigue—F. B. Stulen, presented from manuscript by the author.

Statistical Analysis of Fatigue Data—Robert Plunkett, presented from manuscript by the author.

Prepared Discussions:

E. Epremian, presented from manuscript by the author.

J. T. Ranson, presented from manuscript by the author.

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[5] Issued as separate publication *STP No. 121.*

EIGHTH SESSION—LUNCHEON, PRESIDENT'S ADDRESS, AWARD OF HONORARY MEMBERSHIPS, PRESENTATION OF AWARDS OF MERIT, RECOGNITION OF 40-YEAR MEMBERS, AND INTRODUCTION OF NEW OFFICERS

TUESDAY, JUNE 19, 12:15 P.M.

CHAIRMAN: PAST-PRESIDENT JAMES G. MORROW

About 300 were present at this annual luncheon session, including a number of ladies and visitors.

Presidential Address:

The annual President's Address was presented by L. J. Markwardt, his subject being "An Idea Whose Time Had Come." This interesting address is printed in the July ASTM BULLETIN.

Award of Honorary Memberships:

The Executive Secretary read citations for the following five members who had been elected by the Board of Directors to Honorary Membership in the Society:

Thomas G. Delbridge
Albert T. Goldbeck
Harold H. Morgan
Frank E. Richart
Frederick W. Smither

Mr. Smither was unable to be present at the meeting but the other four received their certificates of Honorary Membership from President Markwardt.

Presentation of Awards of Merit:

The Chairman of the 1951 Award of Merit Committee, L. H. Winkler, read brief citations and presented the following men to President Markwardt, who conferred on them, on behalf of the Board of Directors, the certificate of the Award of Merit:

William Blum
Hyman Bornstein
Robert Burns
H. V. Churchill
Max Hecht
Carl D. Hocker
William H. Klein
H. H. Lester
Stanton Walker
William H. Whitcomb

Recognition of 40-Year Members:

The Executive Secretary read the names of those members, both individual and company, who had been continuously affiliated with the Society for 40 years. Thirteen of the 26 members were present or represented at the meeting, and received the certificates, the other certificates being mailed to the recipients. It is of interest that four branches of the Navy which held memberships in perpetuity were in this group. A list of the 40-year members appears in the preprinted Report of the Board of Directors and the July ASTM BULLETIN.

Election of Officers:

Results of the letter ballot on election of new officers were announced by the Chairman of the Tellers' Committee, H. W. Stuart. The results were as follows:

For President, to serve for one year:
  T. S. Fuller, 1137 votes
For Vice-President, to serve for two years:
  L. C. Beard, Jr., 1133 votes
For Directors, to serve for three years:
  J. W. Bolton, 1134 votes
  R. A. Schatzel, 1131 votes
  E. O. Slater, 1133 votes
  Stanton Walker, 1135 votes
  F. P. Zimmerli, 1134 votes

The newly-elected members of the Board of Directors who were present were introduced, as were President-elect Fuller and Vice-President-elect Beard, the latter two men responding briefly.

Chairman Morrow then presented the Past-President's pin to retiring President L. J. Markwardt. Past-President T. A. Boyd was then introduced, following the custom of having the Senior Past-President who retires from the Board of Directors, recognized. He had completed eight years of service on the Board of Directors.

NINTH SESSION—SYMPOSIUM ON FLAME PHOTOMETRY[4]

(*Continued from Sixth Session*)

TUESDAY, JUNE 19, 2:30 P.M.

SESSION CHAIRMAN: R. O. CLARK

This session was a continuation of the Symposium on Flame Photometry. The papers presented at this session are listed below:

Control of Interferences Caused by Acids and Salts in the Flame Photometric Determination of Sodium and Potassium—F. T. Eggertsen, Garrard Wyld and Louis Lykken, presented from manuscript by Mr. Lykken.

The Effect of Organic Solvents on the Flame

Photometric Emission of Certain Elements—G. W. Curtis, H. E. Knauer, and L. E. Hunter, presented by Mr. Curtis.

Determination of Tetraethyllead in Gasoline by Flame Photometry—P. T. Gilbert, Jr., presented from manuscript by the author.

Determination of Calcium in Lubricating Oils by Flame Spectrophotometer—M. L. Moberg, V. B. Waithman, W. H. Ellis, and H. D. DuBois, presented from manuscript by Mr. Robert Matteson.

A Modified Recording Flame Photometer—W. H. King, Jr., and William Priestley, Jr., presented by Mr. King.

The Flame Photometer in the Analysis of Water and Water-Formed Deposits—R. K. Scott, V. M. Marcy, and J. J. Hronas, presented by Mr. Scott.

TENTH AND ELEVENTH SESSIONS—SYMPOSIUM ON SURFACE AND SUBSURFACE RECONNAISSANCE

Tenth Session

TUESDAY, JUNE 19, 2:30 P.M.
SESSION CHAIRMAN: L. E. GREGG

Committee D-18 on Soils for Engineering Purposes:

Report presented by E. J. Kilcawley, chairman, and the following actions taken:

Adopted as Standard, Revisions in:

Method of Mechanical Analysis of Soils (D 422 - 39)

Symposium on Surface and Subsurface Reconnaissance:[6]

The authors at each of the two sessions served as a panel. Each author covered very briefly the subject matter of his paper, after which the sessions were thrown open to discussion, the authors being asked to answer the various questions raised. The 14 papers comprising the Symposium were presented at the Tenth and Eleventh Sessions:

Interpreting Geologic Maps for Engineers—E. B. Eckel.

Engineering Implications of Geological Reconnaissance in the Plains Area—Missouri River Basin—E. A. Abdun-Nur and J. D. Dowling.

Preliminary Foundation Exploration in Arctic Regions—L. A. Nees and A. M. Johnson.

Preparation of County Engineering Soil Maps for Illinois—T. H. Thornburn.

The Engineer Looks at Pedology—R. L. Greenman.

Application of Aerial Photographs to Preliminary Engineering Soil Surveys—R. D. Miles.

The Preparation of an Engineering Soil Map of New Jersey—D. R. Lueder.

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[6] To be issued as separate publication *STP No. 122.*

Eleventh Session

TUESDAY, JUNE 19, 8 P.M.
SESSION CHAIRMAN: G. W. MCALPIN

Earth Resistivity Tests Applied to Subsurface Reconnaissance Surveys—R. W. Moore.

Electrical Resistivity Method as Applied to Engineering Problems—H. L. Scharon.

Geophysical Measurements of the Depth of Weathered Mantle Rock—Dart Wantland.

The Practical Value of an Earth Resistivity Method in Making Subsurface Explorations—W. F. Abercrombie.

Applications of Seismic Methods to Foundation Exploration—A. M. Johnson and R. H. Wesley.

Seismology Applied to Shallow Zone Research—Daniel Linehan.

Resistivity Reconnaissance—Irwin Roman.

TWELFTH SESSION—FATIGUE

TUESDAY, JUNE 19, 8 P.M.
SESSION CHAIRMAN: T. J. DOLAN

Committee E-9 on Fatigue:

Report presented by R. E. Peterson, chairman, and accepted as a report of progress.

Papers:

Fatigue Strength of Ball Bearing Races and and Heat-Treated 52100 Steel Specimens—Haakon Styri, presented by the author.
Laboratory Evaluation of Materials for Marine Propulsion Gears—M. R. Gross, presented by the author.
The Influence of Surface Roughness on the Fatigue Life and Scatter of Test Results of Two Steels—P. G. Fluck, presented by Mr. E. F. Lundeen.
Effect of Residual Stress on the Fatigue Strength of Notched Specimens—D. Rosenthal and G. Sines, presented by Mr. Sines.
Damping, Elasticity, and Fatigue Properties of Temperature-Resistant Materials—B. J. Lazan and L. J. Demer, presented from manuscript by Mr. Lazan.
Damping, Fatigue, and Dynamic Stress-Strain Properties of Mild Steel—B. J. Lazan and T. Wu, presented by title only.

THIRTEENTH SESSION—METHODS OF TESTING

WEDNESDAY, JUNE 20, 9 A.M.
SESSION CHAIRMAN: PAST-PRESIDENT J. R. TOWNSEND

Committee D-9 on Electrical Insulating Materials:

Report presented by A. H. Scott, chairman, and the following actions taken:

Accepted as Tentative:

Methods of Testing Hydrocarbon Waxes Used as Electrical Insulation

Accepted as Tentative, Revisions in:

Methods of Testing Varnish Used for Electrical Insulation (D 115 - 48 T)
Methods of Testing Varnished Cloths and Varnished Cloth Tapes Used in Electrical Insulation (D 295 - 50 T)
Spec. for Black Bias-Cut Varnished Cloth Tape Used for Electrical Insulation (D 373 - 50 T)
Methods of Testing Nonrigid Polyvinyl Tubing (D 876 - 50 T)
Methods of Testing Sheet and Plate Materials Used in Electrical Insulation (D 229 - 49)
Methods of Testing Pasted Mica Used in Electrical Insulation (D 352 - 49)

Adopted as Standard:

Method of Test for Rockwell Hardness of Plastics and Electrical Insulating Materials (D 785 - 48 T), jointly with Committee D-20 on Plastics
Method of Test for Detection of Free Sulfur in Electrical Insulating Oils (D 989 - 42 T)
Methods of Test for Power Factor and Dielectric Constant of Natural Mica (D 1082 - 49 T)
Rec. Practice for Maintaining Constant Relative Humidity by Means of Aqueous Solutions (D 1041 - 49 T), jointly with Committee D-20 on Plastics

Adopted as Standard, Revisions in:

Methods of Measuring Mica Stamping Used in Electronic Devices and Incandescent Lamps (D 652 - 43)

Withdrawal of Tentatives:

Spec. for Phenolic Laminated Sheet for Radio Applications (D 467 - 44 T)
Spec. for Round Phenolic Laminated Tubing for Radio Applications (D 616 - 41 T)

Reference to the proposed revision of the Standard Method of Test for Power Factor and Dielectric Constant of Electrical Insulating Oils of Petroleum Origin (D 924 – 49) was withdrawn from the report, as preprinted, and this revision will be submitted to the Administrative Committee on Standards.

Committee D-3 on Gaseous Fuels:

Report presented by A. W. Gauger, chairman, and accepted as a report of progress.

Committee D-5 on Coal and Coke:

Report presented by W. A. Selvig, chairman, and the following actions taken:

Adopted as Standard:

Method of Test for Grindability of Coal by the Hardgrove-Machine Method (D 409 – 37 T), as revised

Withdrawal of Tentative:

Method of Test for Grindability of Coal by the Ball-Mill Method (D 408 – 37 T)

Editorial Changes Accepted in:

Method of Test for Size of Anthracite (D 310 – 34)

Committee E-7 on Non-Destructive Testing:

Report presented by J. H. Bly, chairman, and accepted as a report of progress.

Committee A-7 on Malleable-Iron Castings:

Report presented by W. A. Kennedy, chairman, and accepted as a report of progress.

Committee E-1 on Methods of Testing:

Report presented by J. R. Townsend, chairman, and the following actions taken:

Accepted as Tentative, Revisions in:

Methods of Compression Testing of Metallic Materials in Other Than Sheet Form (E 9 – 49 T)
Method of Compression Testing of Metallic Materials in Sheet Form (E 78 – 49 T)
Rec. Practice for Analysis by Microscopical Methods for Particle Size Distribution of Particulate Substances of Subsieve Size (E 20 – 48 T)
Method of Test for Softening Point (Ball and Shouldered Ring Apparatus) (E 28 – 42 T)
Methods of Tension Testing of Metallic Materials (E 8 – 46)

Adopted as Standard, Revisions in:

Spec. for ASTM Thermometers (E 1 - 50)

Papers:

The Constancy of Calibration of Elastic Calibrating Devices—W. C. Aber and F. M. Howell, presented by Mr. Aber.
Mechanical Properties in Torsion and Poisson's Ratio for Certain Stainless Steel Alloys—C. W. Muhlenbruch, V. N. Krivobok, and C. R. Mayne, presented from manuscript by Mr. Muhlenbruch.
An Arbitration Bar Izod Impact Test for Cast Iron—J. T. Eash and A. P. Gagnebin, presented by Mr. Eash.
Electrically Excited Resonant-Type Fatigue Machine—T. J. Dolan, presented from manuscript by the author.[7]

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[7] Published in ASTM BULLETIN, No. 175, July, 1951, p. 60 (TP 150).

FOURTEENTH SESSION—"BALANCING THE ECONOMY FOR DEFENSE PRODUCTION"

WEDNESDAY, JUNE 20, 11:30 A.M.

SESSION CHAIRMAN: PRESIDENT L. J. MARKWARDT

President L. J. Mardwardt introduced Nathaniel Knowles, Deputy Administrator, Defense Production Administration, who discussed the fundamental policies for balancing the production for the military and for the civilian economy, emphasizing the importance of conservation and substitution in strengthening the total defense effort. A discussion period followed the address.

FIFTEENTH SESSION—EFFECT OF TEMPERATURE ON METALS

WEDNESDAY, JUNE 20, 1:45 P.M.

SESSION CHAIRMAN: PAST-PRESIDENT A. E. WHITE

Committee A-1 on Steel:

Report presented by N. L. Mochel, chairman, and the following actions taken:

Accepted as Tentative:

Spec. for Nickel-Chromium-Molybdenum Steel Bars for Springs
Spec. for Alloy Steel Seamless Drum Forgings
Spec. for Seamless and Welded Steel Tubes for Low-Temperature Service
Spec. for Seamless Ferritic Alloy Steel Pipe for High-Temperature Service

It was announced that the committee had withdrawn from the report, as preprinted, the proposed new Tentative Specifications for Carbon-Steel Sheets of Flange and Firebox Qualities.

Accepted as Tentative, Revisions in:

Spec. for General Requirements for Delivery of Rolled Steel Plates, Shapes, Sheet Piling, and Bars for Structural Use (A 6 – 50 T)
Spec. for Boiler Rivet Steel and Rivets (A 31 – 49 T)
Spec. for Seamless Carbon-Steel Pipe for High-Temperature Service (A 106 – 48 T)
Spec. for Seamless Alloy-Steel Pipe for High-Temperature Service (A 158 – 50 T)
Spec. for Heat-Treated Carbon- and Alloy-Steel Track Bolts and Nuts (A 183 – 49 T)
Spec. for Alloy-Steel Bolting Materials for High-Temperature Service (A 193 – 50a T)
Spec. for Nickel-Steel Plates for Boilers and Other Pressure Vessels (A 203 – 50 T)
Spec. for Seamless Carbon-Molybdenum Alloy-Steel Pipe for High-Temperature Service (A 206 – 48 T)
Spec. for Seamless Alloy-Steel Boiler and Superheater Tubes (A 213 – 50 T)
Spec. for Carbon and Alloy Steel Ring and Disk Forgings (A 243 – 49 T)
Spec. for Carbon Steel Seamless Drum Forgings (A 266 – 49 T)
Spec. for Seamless Chromium-Molybdenum Alloy-Steel Pipe for Service at High Temperatures (A 280 – 48 T)
Spec. for Low and Intermediate Tensile Strength Carbon-Steel Plates of Structural Quality (Plates 2 in. and Under in Thickness) (A 284 – 50 T)
Spec. for Chromium-Molybdenum Steel Plates for Boilers and Other Pressure Vessels (A 301 – 50 T)
Spec. for Seamless and Welded Austenitic Stainless Steel Pipe (A 312 – 48 T)
Spec. for Seamless 1 Per Cent Chromium, 0.5 Per Cent Molybdenum Alloy-Steel Pipe for Service at High Temperatures (A 315 – 48 T)
Spec. for Alloy-Steel Bolting Materials for Low-Temperature Service (A 320 – 50 T)
Methods and Def. for Mechanical Testing of Steel Bars (A 330 – 50 T)
Spec. for Oil-Tempered Steel Spring Wire (A 229 – 41)
Spec. for Heat-Treated Carbon-Steel Bolting Material (A 261 – 47)
Spec. for Quenched Carbon-Steel Joint Bars (A 49 – 48)
Spec. for Welded and Seamless Steel Pipe (A 53 – 47)
Spec. for Electric-Resistance-Welded Steel Pipe (A 135 – 46)
Spec. for Electric-Fusion-Welded Steel Pipe (Sizes 4 in. to but Not Including 30 in.) (A 139 – 46)
Spec. for Seamless Low-Carbon and Carbon-Molybdenum Steel Still Tubes for Refinery Service (A 161 – 46)
Spec. for Electric-Resistance-Welded Steel and Open-Hearth Iron Boiler Tubes (A 178 – 47)
Spec. for Seamless Cold-Drawn Low-Carbon Steel Heat-Exchanger and Condenser Tubes (A 179 – 46)
Spec. for Seamless Steel Boiler Tubes for High-Pressure Service (A 192 – 44)
Spec. for Seamless Cold-Drawn Intermediate Alloy-Steel Heat-Exchanger and Condenser Tubes (A 199 – 46)
Spec. for Seamless Intermediate Alloy-Steel Still Tubes for Refinery Service (A 200 – 46)
Spec. for Seamless Carbon-Molybdenum Alloy-Steel Boiler and Superheater Tubes (A 209 – 46)
Spec. for Medium-Carbon Seamless Steel Boiler and Superheater Tubes (A 210 – 46)
Spec. for Electric-Resistance-Welded Steel Heat-Exchanger and Condenser Tubes (A 214 – 47)
Spec. for Electric-Resistance-Welded Steel Boiler and Superheater Tubes for High-Pressure Service (A 226 – 47)
Spec. for Factory-Made Wrought Carbon and Alloy Steel Welding Fittings (A 234 – 50)

Spec. for Electric-Resistance-Welded Carbon-Molybdenum Alloy-Steel Boiler and Superheater Tubes (A 250 - 47)
Spec. for Copper Brazed Steel Tubing (A 254 - 46)

Adopted as Standard:

Spec. for Heat-Treated Steel Leaf Springs (A 147 - 49 T)

Adopted as Standard, Revisions in:

Spec. for Electric-Furnace-Welded Steel Pipe for High-Temperature and High-Pressure Service (A 155 - 42)
Spec. for Steel Music Spring Wire (A 228 - 41)
Spec. for Carbon- and Alloy-Steel Nuts for Bolts for High-Pressure and High-Temperature Service (A 194 - 48)

Withdrawal of Standard:

Spec. for Heat-Treated Steel Elliptical Springs (A 147 - 39)

Committee A-3 on Cast Iron:

Report presented by J. S. Vanick, chairman, and the following action taken:

Accepted as Tentative, Revisions in:

Spec. for Gray Iron Castings for Pressure-Containing Parts for Temperatures Up to 650 F (A 278 - 49 T)

Committee A-5 on Corrosion of Iron and Steel:

Report presented by T. R. Galloway, chairman, and the following actions taken:

Accepted as Tentative:

Spec. for Zinc-Coated Iron or Steel Chain-Link Fence Fabric Galvanized Before Weaving

Accepted as Tentative, Revisions in:

Methods of Test for Local Thickness of Electrodeposited Coatings (A 219 - 45 T), jointly with Committee B-8 on Electrodeposited Metallic Coatings

Adopted as Standard:

Spec. for Zinc-Coated (Galvanized) "Iron" Telephone and Telegraph Line (A 111 - 49 T)

Joint Committee on Effect of Temperature on the Properties of Metals:

Report presented, in the absence of the chairman, by R. J. Painter and accepted as a report of progress.

Papers:

Creep-Rupture of Several Sheet Steels—G. V. Smith, E. J. Dulis, and E. G. Houston, presented from manuscript by Mr. Dulis.
The Fabrication of Chromium-Molybdenum Steel Piping for 1000-1050 F Central Station Service—R. W. Emerson, presented from manuscript by the author.
Microstructural Instability of Steels for Elevated Temperature Service—G. V. Smith, W. B. Seens, H. S. Link and P. R. Malenock, presented by Mr. Smith.
Notch-Toughness of Four Alloy Steels at Low Temperatures—W. B. Seens, W. L. Jensen, and O. O. Miller, presented from manuscript by Mr. Seens.
Apparatus for Low-Temperature Tension Tests of Metals—R. J. Mosborg, presented by the author.[8]
Notch-Toughness of Fully Hardened and Tempered Low-Alloy Steels—R. L. Rickett and J. M. Hodge, presented by Mr. W. B. Seens.

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[8] Published in the ASTM BULLETIN, No. 177, October, 1951, p. 41 (TP 205).

SIXTEENTH SESSION—MARBURG LECTURE; DUDLEY MEDAL AWARD; RICHARD L. TEMPLIN AWARD; SAM TOUR AWARD

WEDNESDAY, JUNE 20, 4 P.M.

SESSION CHAIRMAN: PRESIDENT L. J. MARKWARDT

President Markwardt presented Mr. F. L. LaQue, in charge of Corrosion Engineering Section, International Nickel Co., New York, N. Y., as the Twenty-fifth Marburg Lecturer, who then delivered the lecture on "Corrosion

Testing." Mr. LaQue's lecture comprised a survey of the corrosion-testing programs and the methods of corrosion testing which have been sponsored by ASTM. He particularly discussed the distinction that must be made between the corrodibility of a material and the protective value of its corrosion products and how these are influenced by both the composition of the material and the incidental conditions of its exposure. A few of the topics covered by Mr. LaQue are as follows:

1. Relation between "acid" and other "accelerated" tests and the results of long-time atmospheric corrosion tests.
2. The relationship between the color and other characteristics of rust films and the durabilities of steels in the atmosphere and the prediction of performance on basis of rust color.
3. The influence of the corrosion-resistance of steel on the performance of paint coatings.
4. The use and abuse of results of salt spray tests.
5. Discussion of advantages and limitations of ASTM corrosion methods.

President Markwardt, in expressing appreciation to Mr. LaQue for his very timely and instructive lecture on a subject of particular interest to the ASTM, presented to him, on behalf of the Society, the Edgar Marburg Lecture Certificate.

Medalists and Award Winners:

The Charles B. Dudley Medal, named after the Society's first president and awarded to the author or authors of a paper of outstanding merit constituting an original contribution on research and engineering materials, was presented to D. S. Clark and P. E. Duwez, Associate Professors of Mechanical Engineering, California Institute of Technology, Pasadena, Calif., for their paper on "The Influence of Strain Rate on Some Tensile Properties of Steel," presented at the 1950 Annual Meeting and published in the 1950 *Proceedings*.

The Richard L. Templin Award, to stimulate research in the development of testing methods and apparatus and to encourage the presentation to the Society of papers describing new and useful testing procedures and apparatus, was given to R. L. Templin and W. C. Aber, Aluminum Research Laboratories, Aluminum Company of America, New Kensington, Pa., for their paper on "A Method for Making Tension Tests of Metals Using a Miniature Specimen," presented at the 1950 Annual Meeting and published in the 1950 *Proceedings*. Mr. Aber accepted the award.

The Sam Tour Award, to encourage research on the improvement and evaluation of corrosion testing methods and to stimulate the preparation of technical papers in this field, was given to C. T. Evans Jr., Chief Metallurgist, Elliott Co., Jeannette, Pa., for his paper on "Oil Ash Corrosion of Materials at Elevated Temperatures," presented at the 1950 Annual Meeting and published in the Symposium on Corrosion of Materials at Elevated Temperatures (issued by the Society as *STP No. 108*).

SEVENTEENTH SESSION—CONCRETE

THURSDAY, JUNE 21, 9:30 A.M.

SESSION CHAIRMAN: K. B. WOODS

Committee C-3 on Chemical-Resistant Mortars:

Report presented by F. O. Anderegg, chairman, and the following action taken:

Accepted as Tentative:

Method of Test for Chemical Resistance of Hydraulic Cement Mortars

Committee C-4 on Clay Pipe:

Report presented, in the absence of the chairman, by R. J. Painter and accepted as a report of progress.

Committee C-9 on Concrete and Concrete Aggregates:

Report presented by Kenneth B. Woods, chairman, and the following actions taken:

Accepted as Tentative, Revisions in:

Method of Test for Fundamental Transverse Frequency of Concrete Specimens for Calculating Modulus of Elasticity (Sonic Method) (C 215 - 47 T)

Adopted as Standard, Revisions in:

Method of Test for Abrasion of Coarse Aggregates by Use of the Los Angeles Machine (C 131 - 47), jointly with Committee D-4 on Road and Paving Materials

Sanford E. Thompson Award:

Mr. Woods then presented the ninth Sanford E. Thompson Award to Richard C. Mielenz, Leslie P. Witte, and Omar J. Glantz, Head, Petrographic Laboratory, Head, Durability Laboratory, and Head, Cement Laboratory, respectively, U. S. Bureau of Reclamation, Denver, Colo., for their paper on "Effect of Calcination on Natural Pozzolans" published in the Symposium on Use of Pozzolanic Materials in Mortars and Concretes. This paper was presented at the First Pacific Area National Meeting of the Society held in San Francisco, Calif., October 10–14, 1949. This award was established in 1938 by Committee C-9 on Concrete and Concrete Aggregates as an annual token of recognition to the author or authors of a paper of outstanding merit on concrete and concrete aggregates presented at a meeting of the Society.

Committee C-13 on Concrete Pipe:

Report presented by W. W. Horner, chairman, and the following actions taken:

Accepted as Tentative, Revisions in:

Spec. for Concrete Sewer Pipe (C 14 - 41)
Spec. for Reinforced Concrete Sewer Pipe (C 75 - 41)
Spec. for Reinforced Concrete Culvert Pipe (C 76 - 41)
Spec. for concrete Irrigation Pipe (C 118 - 39)

Papers:

Freeze-Thaw Resistance of Concrete as Affected by Alkalies in Cement—Bailey Tremper, presented by Mr. H. L. Kennedy.
Aggregates Tested by Accelerated Freezing and Thawing of Concrete—W. H. Price and D. G. Kretsinger, presented from manuscript by Mr. Price.
Performance of Automatic Freezing and Thawing Apparatus for Testing Concrete—Stanton Walker and D. L. Bloem, presented from manuscript by Mr. Walker.
Some Properties Affecting the Abrasion Resistance of Air-Entrained Concrete—L. P. Witte and J. E. Backstrom, presented by Mr. R. C. Mielenz.
Permeability Tests of Lean Mass Concrete—H. K. Cook, presented by the author.
Use of the Soniscope for Measuring Setting Time of Concrete—E. A. Whitehurst, presented from manuscript by the author.

EIGHTEENTH SESSION—APPEARANCE, NON-FERROUS METALS, CREEP, CORROSION-RESISTANT ALLOYS

THURSDAY, JUNE 21, 9:30 A.M.

SESSION CHAIRMAN: PAST-PRESIDENT R. L. TEMPLIN

Committee B-6 on Die-Cast Metals and Alloys:

Report presented by J. R. Townsend, chairman, and accepted as a report of progress.

Appendix:

Aluminum Die Castings—The Effect of Process Variables on Their Properties, W. Babington and D. H. Kleppinger, presented by Mr. Babington.

Committee A-10 on Iron-Chromium, Iron-Chromium-Nickel, and Related Alloys:

Report presented, by L. L. Wyman, vice-chairman, and accepted as a report of progress.

Committee B-2 on Non-Ferrous Metal and Alloys:

Report presented by Bruce W. Gonser, chairman, and accepted as a report of progress.

Committee B-7 on Light Metals and Alloys, Cast and Wrought:

Report presented by Walter Bonsack, vice-chairman, and the following actions taken:

Accepted as Tentative, Revisions in:

Spec. for Aluminum-Alloy Sheet and Plate for Use in Pressure Vessels (B 178 - 50 T)
Spec. for Aluminum and Aluminum-Alloy Sheet and Plate (B 209 - 50 T)

Committee E-2 on Emission Spectroscopy:

Report presented by Walter Bonsack, vice-chairman, and accepted as a report of progress.

Committee E-3 on Chemical Analysis of Metals:

Report presented by H. A. Bright, honorary chairman, and accepted as a report of progress.

Committee E-4 on Metallography:

Report presented by L. L. Wyman, chairman, and the following action taken:

Adopted as Standard:

Rec. Practice for Determining the Inclusion Content of Steel (E 45 - 46 T)

The new tentatives, the Proposed Method for Estimating the Average Grain Size of Non-Ferrous Metals and Their Alloys Other Than Copper and Copper-Base Alloys, and the Proposed Rec. Practice for Thermal Analysis of Metals and Alloys, as listed in the preprinted report, were not presented at the Annual Meeting, but are being submitted to the Administrative Committee on Standards for acceptance and publication as tentative.

Committee E-12 on Appearance:

Report presented, in the absence of the chairman, by J. W. Caum and accepted as a report of progress.

Papers:

Creep Properties of Two Tempers of 63S Extruded Aluminum Alloy—O. D. Sherby and J. E. Dorn, presented by F. M. Howell.
Effect of Annealing on the Creep Properties of 2S-O Aluminum Alloy—O. D. Sherby and J. E. Dorn, presented from manuscript by F. M. Howell.
The Creep Properties of Some Forged and Cast Aluminum Alloys—O. D. Sherby, T. E. Tietz, and J. E. Dorn, presented by F. M. Howell.

NINETEENTH SESSION—PLASTICS, PAINT, INDUSTRIAL HYDROCARBONS

THURSDAY, JUNE 21, 2 P.M.
SESSION CHAIRMAN: ROBERT BURNS

Committee D-1 on Paint, Varnish, Lacquer, and Related Products:

Report presented by W. T. Pearce, chairman, and the following actions taken:

Accepted as Tentative:

Spec. for Methanol (Methyl Alcohol)
Spec. for Methyl Isobutyl Ketone
Method of Test for Purity of Acetone and Methyl Ethyl Ketone
Method of Test for Roundness of Glass Spheres
Method of Test for Total Chlorine in Polyvinyl Copolymers Used for Surface Coatings

Accepted as Tentative, Revisions in:

Spec. for Wood to Be Used as Panels in Weathering Tests of Paints and Varnishes (D 358 - 48 T)
Spec. for Dehydrated Castor Oil (D 961 - 48 T)
Method of Conducting Exterior Exposure Tests of Paints on Wood (D 1006 - 49 T)
Method of Chemical Analysis of White Pigments (D 34 - 47)

Adopted as Standard:

Spec. for Red and Brown Iron Oxide Pigments (D 84 - 48 T)
Methods of Chemical Analysis of Dry Cuprous Oxide and Copper Pigments (D 283 - 48 T)
Method of Test for Acetone Extract in Black Pigments (D 305 - 48 T)
Methods of Chemical Analysis of Zinc Yellow Pigment (Zinc Chromate Yellow) (D 444 - 48 T)
Method of Test for 60-deg Specular Gloss of Paint Finishes (D 523 - 49 T)

Methods of Analysis of Diatomaceous Silica Pigment (D 719 – 47 T)
Methods of Evaluating Degree of Resistance of Traffic Paint to Chipping (D 913 – 47 T)
Method of Test for Abrasion Resistance of Coatings of Paint, Varnish, Lacquer, and Related Products by the Falling Sand Method (D 968 – 48 T)
Method of Testing Pure Para Red and Toluene Red Pigments (D 970 – 48 T)
Methods of Measurements of Dry Film Thickness of Paint, Varnish, Lacquer, and Related Products (D 1005 – 49 T)
Methods of Conducting Exterior Exposure Tests of Paints on Steel (D 1014 – 49 T)
Method of Test for Aniline Points and Mixed Aniline Points of Hydrocarbon Solvents (D 1012 – 49 T)

Adopted as Standard, Revisions in:

Spec. for Spirits of Turpentine (D 13 – 34)
Def. of Terms Relating to Paint, Varnish, Lacquer, and Related Products (D 16 – 47)
Methods of Sampling and Testing Turpentine (D 233 – 48)
Spec. for Shellac Varnishes (D 360 – 41)
Spec. for Sampling and Testing Aluminum Powder and Pastes (D 480 – 48)
Spec. for Zinc Dust (Metallic Zinc Powder) (D 520 – 47)
Method of Test for Dry to No-Pick-Up Time of Traffic Paint (D 711 – 48)

Withdrawal of Standard:

Spec. for Lead Titanate (D 606 – 42)

Editorial Changes Accepted in:

Spec. for Iron Blue (D 261 – 47)
Spec. for Ultramarine Blue (D 262 – 47)
Spec. for Copper Phtalocyanine Blue (D 963 – 49)

Accepted for Publication as Information:

Proposed Method of Test for 20-deg Specular Gloss
Proposed Method of Test for 85-deg Sheen

Committee D-16 on Industrial Aromatic Hydrocarbons:

Report presented, in the absence of the chairman, by P. J. Smith, and the following action taken:

Adopted as Standard:

Methods of Test for Specific Gravity of Industrial Aromatic Hydrocarbons (D 891 – 46 T), as revised

Committee D-20 on Plastics:

Report presented by G. M. Kline, chairman, and the following actions taken:

Accepted as Tentative:

Method of Test for Bursting Strength of Round Rigid Plastic Tubing
Method of Test for Measuring Warpage in Sheet Plastics
Method of Test for Apparent Density and Bulk Factor of Granular Thermoplastic Molding Powder

Accepted as Tentative, Revisions in:

Spec. for Melamine-Formaldehyde Molding Compounds (D 704 – 50 T)
Spec. for Molds for Test Specimens of Plastic Molding Materials (D 647 – 50 T)
Def. of Terms Relating to Plastics (D 883 – 49 T), except that the definitions for nylon plastics, polyester plastics, and resin were withdrawn.

Adopted as Standard:

Method of Test for Deformation of Plastics Under Load (D 621 – 48 T)
Method of Test for Rockwell Hardness of Plastics and Electrical Insulating Materials (D 785 – 48 T), jointly with Committee D-9 on Electrical Insulating Materials
Method of Test for Bond Strength of Plastics and Electrical Insulating Materials (D 952 – 48 T)
Method of Measuring Changes in Linear Dimensions of Plastics (D 1042 – 49 T)
Method of Test for Stiffness Properties of Nonrigid Plastics as a Function of Temperature by Means of a Torsional Test (D 1043 – 49 T)
Rec. Practice for Maintaining Constant Relative Humidity by Means of Aqueous Solutions (D 1041 – 49 T), jointly with Committee D-9 on Electrical Insulating Materials

Adopted as Standard, Revisions in:

Rec. Practice for Molding Specimens of Phenolic Materials (D 796 – 49)
Rec. Practice for Molding Specimens of Amino Plastics (D 956 – 50)
Rec. Practice for Determining Temperatures of Standard ASTM Molds for Test Specimens of Plastics (D 958 – 50)
Method of Measuring Shrinkage from Mold Dimensions of Molded Plastics (D 955 – 50)

Withdrawal of Tentative:

Method of Test for Haze of Transparent Plastics by Photoelectric Cell (D 672 – 45 T)

It was announced that the committee had withdrawn from the report, as reprinted, the recommendation that the Recommended Practice for Transfer Molding Specimens of Phenolic Materials (D 1046 - 49 T) be adopted as standard and the recommendation that Tentative Method of Test for Weight Loss of Plastics on Heating (D 948 - 47 T) be withdrawn.

Papers:

Properties of Exposed and Unexposed Polyvinyl Butyral Coated Fabrics—M. I. Landsberg, T. J. DiFilippo, and Ladislav Boor, presented from manuscript by Mr. Landsberg.

Creep Test Methods for Determining Cracking Sensitivity of Polyethylene Polymers—W. C. Ellis and J. D. Cummings, presented by Mr. Ellis.[9]

Creep Relaxation Relations for Styrene and Acrylic Plastics—Joseph Marin and Yoh-Han Pao, presented from manuscript by Mr. Yoh-Han Pao.

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[9] Published in ASTM BULLETIN, No. 178, December 1951, p. 47 (TP 241).

TWENTIETH SESSION—SYMPOSIUM ON MEASUREMENT OF CONSUMER WANTS[10]

THURSDAY JUNE 21, 2 P.M.

HONORARY CHAIRMEN {PENDLETON HERRING
PAST-PRESIDENT H. J. BALL}

SESSION CHAIRMAN: COLONEL ALBERT E. DENNIS

Members of Administrative Committee on Ultimate Consumer Goods

A. G. Ashcroft
R. D. Bonney
A. L. Brassell
Jules Labarthe, Jr.
G. C. MacDonald
H. H. Morgan
P. S. Olmstead

Determination of Soldier Wants—W. C. Schaefer, presented from manuscript by the author.

Determination of Soldiers' Food Wants—R. P. Benedict, presented from manuscript by the author.

Prepared Discussion—D. R. Peryam, presented from manuscript by the author.

(*Symposium continued in Twenty-second Session*)

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[10] To be issued as separate publication *STP No. 117*.

TWENTY-FIRST SESSION—CEMENTITIOUS MATERIALS, ASPHALTS

THURSDAY, JUNE 21, 2 P.M.

SESSION CHAIRMAN: R. R. LITEHISER

Committee C-1 on Cement:

Report presented by W. C. Hanna, vice-chairman, and the following actions taken:

Accepted as Tentative:

Method of Test for Time of Setting of Hydraulic Cement by the Gillmore Needles
Method of Test for Time of Setting of Hydraulic Cement by the Vicat Needles (C 191 - 51 T)
Definition of the term Pozzolan

Accepted as Tentative, Revisions in:

Spec. for Portland-Blast-Furnace Slag Cement (C 205 - 48 T)
Method of Test for Setting Time of Hydraulic Cement in Mortar (C 229 - 49 T)
Method of Test for Fineness of Portland Cement by the Turbidimeter (C 115 - 42)
Methods of Chemical Analysis of Portland Cement (C 114 - 47)

Adopted as Standard:

Method of Test for Fineness of Portland Cement by Air Permeability Apparatus (C 204 - 46 T), as revised

Adopted as Standard, Revision in:

Spec. for Masonry Cement (C 91 - 49)

Committee C-2 on Magnesium Oxychloride and Magnesium Oxysulfate Cements:

Report presented by L. S. Wells, chairman, and the following actions taken:

Adopted as Standard:

Method of Sampling Magnesium Oxychloride Compositions and Ingredients (C 237 – 49 T)
Method of Test for Sieve Analysis of Magnesium Oxychloride Compositions, Aggregates, and Fillers (C 238 – 49 T)
Method of Test for Sieve Analysis of Plastic Calcined Magnesia (C 239 – 49 T)

Committee C-8 on Refractories:

Report presented, in the absence of the chairman, by J. S. Pettibone and the following actions taken:

Accepted as Tentative:

Method of Test for Modulus of Rupture of Castable Refractories
Method of Test for Permanent Linear Change on Firing of Castable Refractories

Accepted as Tentative, Revisions in:

Def. of Terms Relating to Refractories (C 71 – 49)

Adopted as Standard:

Spec. for Castable Refractories for Boiler Furnaces and Incinerators (C 213 – 50 T), as revised

Adopted as Standard, Revisions in:

Methods of Chemical Analysis of Refractory Materials (C 18 – 45)
Def. of Terms Relating to Refractories (C 71 – 49)
Spec. for Refractories for Malleable Iron Furnaces with Removable Bungs, and for Annealing Ovens (C 63 – 47)
Spec. for Refractories for Heavy-Duty Stationary Boiler Service (C 64 – 48)
Spec. for Refractories for Incinerators (C 106 – 48)
Spec. for Refractories for Moderate-Duty Stationary Boiler Service (C 153 – 48)

Committee C-16 on Thermal Insulating Materials:

Report presented, in the absence of the chairman, by J. S. Pettibone and the following actions taken:

Adopted as Standard:

Method of Test for Flexural Strength of Preformed Block Type Thermal Insulation (C 203 – 45 T)

Adopted as Standard, Revisions in:

Def. of Terms Relating to Thermal Insulating Materials (C 168 – 50)

Committee C-18 on Natural Building Stones:

Report presented, in the absence of the chairman, by J. S. Pettibone and the following action taken:

Adopted as Standard:

Method of Test for Abrasion Resistance of Stone Subjected to Foot Traffic (C 241 – 50 T), as revised

Committee C-21 on Ceramic Whitewares:

Report presented, in the absence of the chairman, by J. S. Pettibone and accepted as a report of progress.

Committee C-22 on Porcelain Enamel:

Report presented, in the absence of the chairman, by J. S. Pettibone and accepted as a report of progress.

Committee D-4 on Road and Paving Materials:

Report presented by C. E. Proudley, chairman, and the following actions taken:

Accepted as Tentative, Revisions in:

Def. of Terms Relating to Materials for Roads and Pavements (D 8 – 49)
Spec. for Emulsified Asphalt (D 977 – 49)

Adopted as Standard:

Spec. for Bituminous Mixing Plant Requirements (D 995 – 49 T)
Methods of Sampling Bituminous Paving Mixtures (D 979 – 49 T)
Rec. Practice for Bituminous Mixing Plant Inspection (D 290 – 49 T)

Adopted as Standard, Revisions in:

Def. of Terms Relating to Materials for Roads and Pavements (D 8 – 49)
Method of Test for Abrasion of Coarse Aggregate by Use of the Los Angeles Machine (C 131 – 47), jointly with Committee C-9 on Concrete and Concrete Aggregates

Committee D-8 on Bituminous Waterproofing and Roofing Materials:

Report presented by A. J. Steiner, chairman, and the following actions taken:

Accepted as Tentative:

Methods of Testing Asphalt-Base Emulsions for Use as Protective Coatings for Built-Up Roofs, changing the words "nonvolatile components" to read "residue by evaporation" in Sections 7 and 9.

Adopted as Standard:

Spec. for Sieve Analysis of Granular Mineral Surfacing for Asphalt Roofing and Shingles (D 1001 - 48 T)

Adopted as Standard, Revisions in:

Spec. for Asphalt Shingles Surfaced with Mineral Granules (D 225 - 46)

Spec. for Wide Selvage Asphalt Roofing Surfaced with Mineral Granules (D 371 - 46)

Spec. for Asphalt Siding Surfaced with Mineral Granules (D 699 - 46)

Editorial Changes Accepted in:

Methods of Sampling and Testing Felted and Woven Fabrics Saturated with Bituminous Substances for Use in Waterproofing and Roofing (D 146 - 47)

Papers:

Changes in Characteristics of Portland Cement as Exhibited by Laboratory Tests Over the Period 1904 to 1950—H. F. Gonnerman and William Lerch, presented from manuscript by Mr. Gonnerman.[11]

Report on Further Studies to Develop an Accelerated Test Procedure for the Detection of Adversely Reactive Cement-Aggregate Combinations—T. E. Stanton, presented from manuscript by Mr. H. F. Gonnerman.

Method of Making Vibrated Dry-Tamp Concrete Cylinders Applied to Tests of Lightweight Aggregates and Block Mixtures—S. B. Helms and A. L. Bowman, presented from manuscript by Mr. Helms.

A Study of Aged White Coat Plaster by Differential Thermal Analysis—J. A. Murray and H. C. Fischer, presented by Mr. Murray.

Progressive Heterogeneity on Aging, in Naphtha Sols of "Negative Spot Test" Asphalts—G. L. Oliensis, presented from manuscript by the author.

TWENTY-SECOND SESSION—SYMPOSIUM ON MEASUREMENT OF CONSUMER WANTS[10]

(*Continued from Twentieth Session*)

THURSDAY, JUNE 21, 8 P.M.

SESSION CHAIRMAN: PAST-PRESIDENT H. J. BALL

This session was a continuation of the Symposium on Measurement of Consumer Wants. The papers presented at this session are listed below:

The General Problem of Measurement—S. A. Stouffer, presented from manuscript by the author.

Interviewer Bias—Clyde Hart, presented from manuscript by the author.

Some Application of the Panel Technique in Social Research—C. Y. Glock, presented from manuscript by the author.

Effective Sampling Procedures—F. F. Stephan and P. J. McCarthy, presented from manuscript by Mr. Stephan.

TWENTY-THIRD SESSION—SYMPOSIUM ON STRUCTURAL SANDWICH CONSTRUCTION[12]

THURSDAY, JUNE 21, 8 P.M.

SESSION CHAIRMAN: D. G. REID

Developments and Trends in Lightweight Composite Construction—L. J. Markwardt, presented from manuscript by the author.

Sandwich Construction in the Elastic Range—H. W. March, presented from manuscript by Mr. C. B. Norris.

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[11] Issued as separate publication *STP No. 127.*

[12] Issued as separate publication *STP No. 118.*

Strength of Sandwich Construction—C. B. Norris, presented by the author.

Compressive and Torsional Instability of Sandwich Cylinders—George Gerard, presented by the author.

Paper Honeycomb as a Core for Structural Sandwich Construction—E. W. Kuenzi, presented from manuscript by the author.

Aluminum Honeycomb Sandwich Construction—T. P. Pajak, presented by the author.

Some Developments in Structural Sandwich Building Panels Having Inorganic Cores—G. M. Rapp, presented by the author.

Fabrication Techniques for Structural Sandwich Constructions—B. G. Heebink, presented from manuscript by the author.

TWENTY-FOURTH SESSION—SYMPOSIUM ON ACOUSTICAL MATERIALS[13]

FRIDAY, JUNE 22, 9:30 A.M.

SESSION CHAIRMAN: H. A. LEEDY

Brief History of the Acoustical Materials Industry—Wallace Waterfall, presented from manuscript by the author.

Activity of Committee C-20—H. A. Leedy, presented from manuscript by the author.

The Measurement of Sound Absorption—H. J. Sabine, presented from manuscript by the author.

Combustibility of Acoustical Materials—Wallace Waterfall, presented from manuscript by the author.

Maintenance of Acoustic Materials—Peter Chrzanowski and Albert London, presented from manuscript by Mr. London.

The Application of Acoustical Materials—L. F. Yerges, presented from manuscript by the author.

Basic Physical Properties of Acoustical Materials—William Jack, presented from manuscript by the author.

Committee C-20 on Acoustical Materials:

Report presented by H. A. Leedy, chairman, and accepted as a report of progress.

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[13] To be issued as separate publication *STP No. 123.*

TWENTY-FIFTH SESSION—REPORT SESSION

FRIDAY, JUNE 22, 11:30 A.M.

SESSION CHAIRMAN: PRESIDENT L. J. MARKWARDT

Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys:

Reported presented, in the absence of the chairman, by J. S. Pettibone and accepted as a report of progress.

Committee D-6 on Paper and Paper Products:

Report presented by W. R. Willets, chairman, and the following actions taken:

Accepted as Tentative:

Method of Test for Chloride Content of Paper and Paper Products

Method of Test for Water-Soluble Matter in Paper

Method of Test for Lint of Paper Towels

Method of Test for Ring Crush of Paperboard, with the following changes:

Section 1.—Revise the second sentence to read as follows with the addition of the italicized words and the deletion of those in brackets: "Corrugated and solid fiberboard containers are subjected to crushing forces in shipment. [and] This test is used for two purposes *in the evaluation of the paperboard to be used as components of such fiberboard containers:* (*1*) to indicate edgewise rigidity of the board so that manufacturing processes may be controlled to secure the desired results, and (*2*) to indicate the probable crushing resistance of the finished container.

Section 2(*a*).—Revise item (*1*) to read as follows by the addition of the italicized words and the deletion of those in brackets: "(*1*) an upper and a lower platen, one of which may be fixed and the other movable [and both held rigidly parallel]. *The surface of the platens shall remain smooth, flat, and parallel to each other within one part in two thousand throughout the test and shall be so mounted as to have not more than 0.002 in. movement in the horizontal direction.*"

Section 2(*b*).—In the first sentence change the limits of the annular groove to read "0.25

± 0.01 in. deep and 1.94 ± 0.001 in. outside diameter." Also add a second sentence to read "The base of the annular groove shall be parallel with the base of the block to ±0.0005 in."

Section 2(c).—Revise the third sentence by addition of the italicized words to read "The preferred method of accomplishing this is by mean of a *punch and die, or lacking this, a* card cutter equipped with an adjustable outside guide. Add a new sentence to read "The cutting device shall be so operated as to cut the specimen with long edges parallel to one part in ten thousand."

Section 3.—In the fifth sentence add to item (*1*) "to one part in ten thousand". Add a new item (*4*) to read "(*4*) The long edges are cut clean without wipe or being torn or frayed in any way by poorly functioning cutting equipment."

Accepted as Tentative, Revisions in:

Methods of Testing Analytical Filter Papers (D 981 - 48 T)
Methods of Test for Water-Vapor Permeability of Paper and Paperboard (D 988 - 48 T)

Adopted as Standard:

Methods of Creasing Paper for Permeability Tests (D 1027 - 49 T)
Method of Test for Ply Separation of Combined Container Board (D 1028 - 49 T)

Committee D-7 on Wood:

Report presented by President L. J. Markwardt, chairman, and the following actions taken:

Accepted as Tentative:

Method of Test for Methoxyl Groups in Woods
Nomenclature of Domestic Hardwoods and Softwoods (D 9 - 51T), with editorial corrections

Adopted as Standard:

Spec. for Creosoted End-Grain Wood Block Flooring for Interior Use (D 1031 - 49 T)

Committee D-10 on Shipping Containers:

Report presented, in the absence of the chairman, by J. S. Pettibone and the following actions taken:

Adopted as Standard:

Method of Test for Water-Vapor Permeability of Packages (D 895 - 47 T)
Method of Test for Water Resistance of Containers by the Spray Method (D 951 - 47 T)
Method of Test for Penetration of Liquids into Submerged Containers (D 998 - 48 T)
Methods of Test for Vapor Permeability of Shipping Containers (D 1008 - 49 T)

Accepted for Publication as Information:

Proposed Method of Tests for Pallets

Committee D-12 on Soaps and Other Detergents:

Report presented, in the absence of the chairman, by J. S. Pettibone and the following actions taken:

Accepted as Tentative:

Method of Test for pH of Aqueous Solutions of Soaps and Detergents
Method of Test for Foaming Properties of Surface-Active Agents

Accepted as Tentative, Revisions in:

Spec. for Chip or Granular Soap for Low-Temperature Washing (Low and Medium Titer) (D 1111 - 50 T)
Spec. for Solid Soap for Low-Temperature Washing (Low and Medium Titer) (D 1112 - 50 T)

Adopted as Standard, Revisions in:

Spec. for Chip Soap (D 496 - 39)
Spec. for Powdered Soap (Nonalkaline Soap Powder) (D 498 - 39)
Spec. for Liquid Toilet Soap (D 799 - 45)
Def. of Terms Relating to Soaps and Other Detergents (D 459 - 48)

Editorial Changes Accepted in:

Methods of Sampling and Chemical Analysis of Soaps and Soap Products (D 460 - 46)

Accepted for Publication as Information Only:

Proposed Methods of Test for Surface and Interfacial Tension of Solutions of Surface-Active Agents
Proposed Method of Test for Total Immersion Corrosion Test for Soak Tank Metal Cleaners
Proposed Method of Test for Rinsing Properties of Metal Cleaners
Proposed Method of Test for Buffering Action of Metal Cleaners

Committee D-13 on Textile Materials:

Report presented, in the absence of the chairman, by the past-chairman H. J. Ball, and the following actions taken:

Accepted as Tentative:

Methods of Test for Resistance to Abrasion of Textile Fabrics, with the words "by rubbing against another surface" added to the definition of "abrasion."

Accepted as Tentative, Revisions in:

Def. of Terms Relating to Textile Materials (D 123 – 50 T)

Spec. and Methods of Test for Asbestos Yarns (D 299 – 50 T)

Spec. and Methods of Test for Asbestos Lap (D 1061 – 50 T)

Method of Test for Apparent Fluidity of Dispersions of Cellulose Fibers (D 539 – 48 T)

Spec. and Methods of Tests for Asbestos Tape for Electrical Purposes (D 315 – 49)

Spec. and Methods of Tests for Asbestos Roving for Electrical Purposes (D 375 – 49)

Methods of Testing Woven Asbestos Cloth (D 577 – 42)

Methods of Testing Asbestos Tubular Sleeving (D 628 – 44)

Methods of Testing Felt (D 461 – 50)

Methods of Testing and Tolerance for Single Jute Yarn (D 541 – 49)

Adopted as Standard:

Def. of Terms Relating to Textile Materials (D 123 – 50 T)

Spec. for Woven Cotton Tapes for Electrical Purposes (D 335 – 48 T)

Method of Test for Stretch of Hosiery (D 1058 – 49 T)

Methods of Testing Single Kraft Yarn (D 1057 – 49 T)

Spec. for Mechanical Sheet Felt (D 1114 – 50 T)

Adopted as Standard, Revisions in:

Def. of Terms Relating to Textile Materials (D 123 – 50)

Methods of Testing Felt (D 461 – 50)

Withdrawal of Tentative:

Spec. for Bleached Cotton Broadcloth (D 504 – 41 T)

Withdrawal of Standard:

Spec. for Terry (Turkish) Toweling (D 505 – 40)

Editorial Changes Accepted in:

Spec. and Methods for Asbestos Yarns (D 299 – 50 T)

Spec. and Methods of Test for Asbestos Roving for Electrical Purposes (D 375 – 49)

Spec. for Woven Asbestos Cloth (D 677 – 50)

Committee D-14 on Adhesives:

Report presented by Frank W. Reinhart, chairman, and the following actions taken:

Accepted as Tentative:

Method of Determining the Effect of Bacterial Contamination on the Permanence of Adhesive Preparations and Adhesive Bonds, with a revision in Section 9, changing the word "supplier" to read "manufacturer" and a correction in the note, changing the address of the American Type Culture Collection to 2029 M St., N.W., Washington 6, D. C.

Adopted as Standard:

Method of Test for Resistance of Adhesive Bonds to Chemical Reagents (D 896 – 50 T)

Method of Test for Applied Weight Per Unit Area of Dried Adhesive Solids (D 898 – 47 T)

Method of Test for Applied Weight Per Unit Area of Liquid Adhesive (D 899 – 47 T)

Method of Test for Cleavage Strength of Metal-to-Metal Adhesives (D 1062 – 49 T)

Committee D-15 on Engine Antifreezes:

Report presented by H. R. Wolf, chairman, and the following actions taken:

Accepted as Tentative:

Method of Test for Freezing Point of Aqueous Engine Antifreeze Solutions

Method of Sampling and Preparing Aqueous Engine Antifreeze Solutions

Accepted as Tentative, Revisions in:

Method of Test for Boiling Point of Engine Antifreeze (D 1120 – 50 T)

Committee D-17 on Naval Stores:

Report presented by V. E. Grotlisch, chairman, and the following actions taken:

Accepted as Tentative:

Def. of Terms Relating to Naval Stores and Related Products (D 804 – 51 T) with revised definitions of "ester gum" and "Gloss Oil"

Accepted as Tentative, Revisions in:

Method of Test for Unsaponifiable Matter in Rosin (D 1065 – 49 T),

Adopted as Standard:

Method of Test for Tall Oil (D 803 – 49 T) with the following changes:

Section 32 (a).—Delete the words "or with a 35/20 ball joint."

Sections 33, 38 and 59 (a).—Add the words "using ethyl alcohol" after the words "Section 23."

Section 60.—Revise this section to read as follows:

60. (*a*) Transfer 5.00 g of the sample to a small Erlenmeyer flask, add 12 to 15 ml of the alkali solution and reflux for 1½ hr. Remove the flask from the condenser and add 50 ml of distilled water. Transfer carefully to a 500-ml separation funnel. Rinse the flask with 40 ml of ether, then pour the ether into the separatory funnel. Shake the funnel well and allow to stand until the ether layer separates. Draw off the aqueous soap solution (lower layer) into a second separatory funnel, allowing a few drops of the water layer to remain above the stopcock to prevent loss of the ether layer by creep through the ground-glass joint.

(*b*) To the soap solution in the second separatory funnel, add 30 ml of ether and extract as described in Paragraph (*a*), drawing off the soap layer into the original saponification flask. Add the ether in the second funnel to the ether in the first funnel; then pour the soap solution in the flask into the second separatory funnel and extract again with 30 ml of ether. Draw the soap layer off into the flask again and add the ether layer to the first separatory funnel as described above. At this time draw off all but a few drops of the soap solution which has collected below and combined ether layers in the first funnel and add to the soap solution before the final ether extraction.

(*c*) Extract the combined soap solution for the fourth time with 30 ml of ether and again add the ether layer to the first separatory funnel. The soap layer may now be discarded.

(*d*) To the first separatory funnel containing the four combined ether extracts, add 2 ml of distilled water after first drawing off any soap solution that may have collected above the stopcock. Then shake the funnel and draw off the water, which is discarded. Repeat this washing, first with 5 ml and then with 30 ml of water.

(*e*) Draw off the washed ether extracts into a dry tared evaporating dish, rinsing the funnel with about 10 ml of ether. Evaporate the ether on a steam bath. After the ether has evaporated, place the dish in an oven at 105 C and dry to constant weight. This usually requires 10 to 15 min. If any droplets of water are observed in the dish, add a few ml of ethyl alcohol and again heat on a steam bath and in the oven until a clean, dry residue is obtained.

Sections 32 (a), 39 (a), 44 (a), 49 (a) and 54 (a).—Change the size of the flask from "300 ml" to "250 ml".

Method of Test for Ash in Rosin (D 1063 – 49 T)

Method of Test for Iron in Rosin (D 1064 – 49 T)

Method of Test for Acid Number of Rosin (D 465 – 49 T), with Sections 9 (*c*) and (*d*) further revised as follows:

(*c*). Titrate with the standard alkali, recording buret and pH meter readings. Sufficient alkali may be added initially to bring the pH of the solution to about 8.0. Allow sufficient time for the electrode system to reach equilibrium. Add alkali in 1.0 ml portions until the change in pH per increment added amounts to about 0.3 pH units. Reduce the additions of alkali to 0.1 ml until the end point has been passed, as indicated by a significant decrease in pH per 0.1 ml added. Continue the titration with 1.0 ml portions until it becomes apparent that the inflection point has been well defined.

(*d*) Determine the inflection point (point of maximum change in pH per ml of alkali solution) to the nearest 0.1 ml by plotting pH readings against milliliters of alkali used. For greater accuracy a plot may be made of the change in pH per ml of alkali against the pH. The peak of this curve will indicate the exact inflection point. The inflection point shall be taken as the end point of the titration.

Method of Test for Saponification Number of Rosin (D 464 – 49 T), with Sections 10 (*d*) and 10 (*e*) revised like Sections 9 (*c*) and 9 (*d*) of Method D 465 above, referring to acid solution and pH of 12.0.

Method of Test for Water in Liquid Naval Stores (D 890 – 50 T), as revised

It was announced that the committee withdrew from the report as preprinted the recommendation for adoption as standard of the Tentative Method of Test for Unsaponifiable Matter in Rosin (D 1065 – 49 T). The method is being retained in its tentative status with the revisions given in the preprinted report.

Committee D-21 on Wax Polishes and Related Material:

Report presented from manuscript, in the absence of the chairman, by J. S. Pettibone and accepted as a report of progress.

Committee C-7 on Lime:

Report presented, in the absence of the chairman, by J. S. Pettibone and accepted as a report of progress.

Committee C-15 on Manufactured Masonry Units:

Report presented by J. W. Whittemore, chairman, and the following actions taken:

Adopted as Standard:

Spec. for Vitrified Clay Filter Block for Trickling Filters (C 159 - 48 T)

Adopted as Standard, Revisions in:

Spec. for Sand-Lime Building Brick (C 73 - 39)

Editorial Changes Accepted in:

Spec. for Clay Building Brick (C 62 - 50)
Methods of Sampling and Testing Structural Clay Tile (C 112 - 36)

Committee C-19 on Structural Sandwich Constructions:

Report presented, in the absence of the chairman, by J. S. Pettibone and the following actions taken:

Accepted as Tentative:

Method of Test for Water Absorption of Core Materials for Structural Sandwich Construction
Method of Test for Density of Core Materials of Structural Sandwich Construction
Method of Shear Test of Sandwich Constructions in Flatwise Plane
Def. of Terms Relating to Structural Sandwich Construction

It was announced that the committee had withdrawn the recommendation that the proposed Methods of Test for Compressive Properties of Structural Sandwich Constructions be accepted for publication as tentative.

Committee D-11 on Rubber and Rubber-Like Materials:

Report presented by Simon Collier, chairman, and the following actions taken:

Accepted as Tentative:

Spec. for Gasket Materials for General Automotive and Aeronautical Purposes
Method of Test for Weather Resistance Exposure of Automotive Rubber Compounds

Accepted as Tentative, Revisions in:

Method of Test for Tension Testing of Vulcanized Rubber (D 412 - 49 T)
Methods of Testing Rubber Adhesives (D 816 - 46 T)
Method of Measuring Low-Temperature Stiffening of Rubber and Rubber-Like Materials by the Gehman Torsional Apparatus (D 1053 - 49 T)

Committee C-12 on Mortars for Unit Masonry:

Report presented, in the absence of the chairman, by J. S. Pettibone and the following action taken:

Accepted as Tentative:

Spec. for Mortar for Unit Masonry

Committee D-19 on Industrial Water:

Report presented by R. E. Hess, Assistant Executive Secretary, and the following actions taken:

Accepted as Tentative:

Method of Test for Chloroform-Extractable Matter in Industrial Water
Methods of Test for Fluoride Ion in Industrial Water, with editorial changes

Accepted as Tentative, Revisions in:

Methods of Test for Acidity and Alkalinity in Industrial Water (D 1067 - 49 T)

Adopted as Standard:

Method of Test for Iron Bacteria in Industrial Water (D 932 - 47 T)
Methods of Test for Sulfate-Reducing Bacteria in Industrial Water and Water-Formed Deposits (D 993 - 48 T)

Adopted as Standard, Revisions in:

Def. of Terms Relating to Industrial Water (D 1129 - 50)

Committee D-2 on Petroleum and Petroleum Products:

Report presented by W. T. Gunn, secretary, and the following actions taken:

Accepted as Tentative:

Method of Test for Total Inhibitor Content (p-Tertiary-Butycatechol) of Butadiene
Method of Test for Bromine Number of Petroleum Distillates (Color Indicator Method)
Method of Test for Bromine Number of Petroleum Distillates (Electrometric Method)

Method of Test for Reduced Pressure Distillation of Petroleum Products

Accepted as Tentative, Revisions in:

Method of Test for Distillation of Plant Spray Oils (D 447 – 41)
Method of Test for Unsulfonated Residue of Plant Spray Oils (D 483 – 40)
Method of Test for Carbon Residue of Petroleum Products (Ramsbottom Coking Method) (D 524 – 42)
Method of Test for Steam Emulsion of Lubricating Oils (D 157 – 42)[14]
Spec. for Aviation Gasoline (D 910 – 50 T), with the inclusion of requirements for Grade 80–87. The committee withdrew from the report the requirements for the new proposed Grade 108–135 as preprinted.
Spec. for Diesel Fuels (D 975 – 50 T)
Method of Test for Aniline Points and Mixed Aniline Points of Petroleum Products (D 611 – 47 T)
Method of Test for Oil Content of Paraffin Wax (D 721 – 49 T)
Method of Test for Chlorine in Lubricating Oils and Greases by the Bomb Method (D 808 – 50 T)
Method of Test for Aromatic Hydrocarbons in Olefin-Free Gasolines by Silica Gel Adsorption (D 936 – 49 T)
Method of Test for Evaporation Loss of Lubricating Greases and Oils (D 972 – 48 T)
Methods of Test for Neutralization Value (Acid and Base Numbers) by Color-Indicator Method (D 974 – 48 T)
Method of Test for Determinations of Purity from Freezing Points (D 1016 – 50 T)
Method of Test for Boiling Point Range of Polymerization-Grade Butadiene (D 1088 – 50 T)

The recommendation that Specifications for Gasoline (D 439 – 50 T) be revised was withdrawn by the committee.

Adopted as Standard:

Method of Test for Sulfated Residue from New Lubricating Oils (D 874 – 47 T)
Method of Test for Knock Characteristics of Motor Fuels by the Research Method (D 908 – 49 T)
Method of Test for Benzene and Toluene by Ultraviolet Spectrophotometry (D 1017 – 49 T)
Method of Test for Total Olefinic Plus Aromatic Hydrocarbons in Petroleum Distillates (D 1019 – 49 T)
Method of Test for Apparent Viscosity of Lubricating Greases (D 1092 – 50 T)
Method of Test for Sodium in Lubricating Oils and Lubricating Oil Additives (D 1026 – 49 T), as revised

Adopted as Standard, Revisions in:

Method of Test for Flash Point by Means of the Tag Closed Tester (D 56 – 36)
Method of Test for Sulfur in Petroleum Products and Lubricants by the Bomb Method (D 129 – 50)
Def. of Terms Relating to Petroleum (D 288 – 49), with the deletion of the definition for "lubricating grease", and the deletion of the second sentence in the Note under definition of "Petroleum Spirits"
Method of Test for Neutralization Value (Acid and Base Number) by Electrometric Titration (D 664 – 49)

Accepted for Publication as Information Only:

Proposed Method for Penetration of Lubricating Greases Worked More Than 60 Strokes
Proposed Method of Test for Measurement of Density and Specific Gravity of Liquids to the Fifth Place (Bingham Pycnometer Method)
Proposed Color-Indicator Test for Saponification Number of Petroleum Products (Para-Xylenol Blue Color-Indicator Method)
Proposed Method of Test for Oil Content of Petroleum Waxes
Proposed Method of Test for Kinematic Viscosity
Proposed Method of Test for Motor and Research Octane Numbers of Small Samples
Proposed Method of Test for Motor and Research Octane Numbers of Small Samples by Multi-Pipet System

Editorial Revisions Accepted in:

Methods of Analysis of Grease (D 128 – 47), with a change in Section 1 on Scope
Method of Test for Oxidation Stability of Lubricating Greases by the Oxygen Bomb Method (D 942 – 50), rewording Section 6 (*d*)

The report of Sectional Committee Z-11 on Petroleum Products and Lubricants, not on the program, was presented by W. T. Gunn, assistant secretary of the committee, and received as information. The recommendations in the report dealing with approvals of American Standards by the American Standards Association will be presented to the Administrative Committee on Standards in accordance with established procedure.

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[14] This recommendation, not preprinted, was accepted subject to unanimous affirmative letter ballot of Committee D-2, which ballot has been favorable.

"AN IDEA WHOSE TIME HAD COME"

ANNUAL ADDRESS BY THE PRESIDENT

L. J. MARKWARDT[1]

JUNE 19, 1951

VICTOR HUGO once said, "No army is as powerful as an idea whose time has come."

When the ASTM was organized some 50 years ago, it was predestined to be "an idea whose time had come." Conceived as a medium for promoting knowledge of materials, for developing specifications for materials, and for standardizing methods of test, the idea fell on fertile soil at the beginning of a new era. That the ASTM has become of age, with a membership now past the 7000 mark, and with some 1800 standards comprising six volumes of 8500 pages, is now history. It is interesting to review the conditions and the principles that have enabled the Society to assume its significant and pre-eminent place in the national economy, and to appraise the tenets that must serve as a guide to an even more important role in the future.

At the turn of the century, when the ASTM was founded, the foundations had only recently been laid for our great industrial expansion and development. The pioneering era—with its need of individual self-sufficiency—had come to a close. The invention of the reaper—and other mechanized equipment—was already on its way to improve the efficiency of agriculture and industry, to reduce almost completely the drudgery and limitations of hand operations, and to set the stage for unprecedented mass production.

BASIC FACTORS IN AMERICAN DEVELOPMENT

America was eminently fortunate in having a wealth of natural resources and conditions to invite development—fertile lands and prairies; the finest stands of virgin forests the world had ever known; abundant waters and streams; invaluable iron ore and other varied mineral deposits; rich coal and oil resources and not last nor least, a temperate, varied, stimulating, and healthful climate favorable to agriculture and to human achievement. Among other elements present in the American formula for progress were the indomitable spirit of the composite population; a system of government based on freedom of thought that fostered free enterprise, with due reward for initiative and incentive. There was also a recognition of the value of standardization and the techniques of mass production.

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[1] Assistant Director, U.S. Forest Products Laboratories, Madison, Wis.

TODAY'S AMERICA

As nations go, our country is still very young. One hundred seventy-five years ago we were a struggling group of colonies with a population of about 2,500,000. By 1900, we had grown to 76,000,000. Today we find ourselves the strongest, most prosperous nation in the world, with the highest standard of living ever known. We have only 6 per cent of the world's land and 7 per cent of the world's population. This 7 per cent owns 72 per cent of the world's automobiles, 50 per cent of the world's radios and telephones. This 7 per cent uses half of the world's rubber and coffee, two thirds of the world's oil and silk, and produces 34 per cent of the world's meat and 47 per cent of all manufactured goods in the world. This is the record made by the American free enterprise system, with the benefits of the abundant natural resources.

CHALLENGE OF THE FUTURE

Just as we of this generation have inherited the America of yesterday, so today's America, with all its developments and resources, must inevitably be taken over by the younger generation. Someone has said: "What our forefathers have bequeathed to us, we must earn to possess." What is done to earn this heritage will determine in large measure the future of America.

One virtue that would not come amiss is modesty. It is not to be unexpected that a wealth of production of consumer goods, and an unprecedented standard of living with innumerable labor-saving devices, may invite envy. There is an old proverb that great prosperity and modesty seldom go together. Prosperity needs no apology, but the need has been suggested for employing wisdom and discretion, generosity, and grace in dealing with this heritage. This is significant also because it is difficult to recount the many luxuries now regarded as essentials without the appearance of boasting. An old couplet may be apropos:

"To earn respect, and not dismay.
Boast only in silence, without display."

The challenge of the future is naturally the preservation and further development of those basic factors that have comprised the essential ingredients in the formula of American economy and way of life:

The courage, energy, resourcefulness, imagination, and spirit of its people—
the system of government that permits great individual freedom—
the incentive motive—
the conservation of natural resources, and
the benefits of standardization and mass production.

To earn this heritage we must guard the American formula and protect it from enemies both within and without. The system is not perfect—no system is perfect—it has weaknesses that require continued changes and repair. In other words, we cannot take this system and this freedom as a matter of course, or we shall lose it. We must continue to be willing to earn it by fighting for it. A great nation dare not become static. It must either move forward or decline. And we are reminded by Oliver Goldsmith that even wealthy countries may decline, when he says:

> "Ill fares the land to hastening ills a prey.
> Where wealth accumulates and men decay."

Role of the Incentive Motive

While all the basic elements in the formula for progress are important, it may be appropriate to single out a few for special comment. Among these the significance of the incentive motive cannot be overemphasized.

Incentive thrives on opportunity. The stimulation of achievement by reward provides incentive, and with incentive much can be accomplished individually and collectively. Remove the incentive motive and progress is at once retarded if not stalemated.

The Amana Community:

Located in Iowa, in a charming valley from which the surrounding hills recede like the steps of a Greek Theater, is the Amana community. Seven old-fashioned villages nestle on the hillsides, surrounded by some 26,000 acres of fertile farmland.

Amana was founded nearly a century ago. Much has been written about it. Its history gives an interesting example of the importance of the incentive motive even in a communal enterprise. The story in brief, as abstracted from *Fortune* (December, 1949), is as follows:

"How would they set up the community? ...Henceforth, no one would have more than anyone else; the land, they agreed, would 'remain a common estate and property...as also with all the labor, cares...and burdens, of which each member shall bear his alloted share with a willing heart.' Control, spiritual and temporal, would rest with thirteen trustees....

"The fruit of their labors they turned over to the common storehouse, and though one might produce more than another, all were rewarded equally.... Soon the new community was prospering.... Year after year prosperity continued.

"Somehow it happened.... As third generation succeeded second, skepticism set in; there was more grumbling and discontent, and the idealism ..began to disappear.... Once again the world had intruded.... 'World clothes' appeared in Amana closets. Musical instruments were smuggled in. A trustee bought a car. Without the old spirit to guide them, they became more occupied with their benefits than with the common effort that provided them. Many grew slothful in their work....

"Each year productivity declined more rapidly. Fixed charges, however, remained the same, and in the twenties, for the first time in Amana history, red ink crept into the colony's books. By 1929 the surplus had almost vanished, and when the depression came to send farm prices plummeting, annual deficits began mounting disastrously. By 1931 they reached $500,000, and for the trustees catastrophe could no longer be ignored. The colony was about to go bankrupt.

"What to do. Shocked out of their lethargy, the members selected a committee of forty-seven to study the situation. After months of debate and emergency town meetings, the committee came up with its recommendation. It was drastic...and on February 1, 1932, by a nine to one margin, the members voted a new constitution. Two months later...the new era began. Amana had gone capitalistic.

"Under the new constitution, religious and temporal authority were separated and the society set up a sort of stock cooperative. Land, buildings, livestock, and all other assets were appraised at $2,200,000, and with this as net capital a corporation was formed.... There were still some benefits...but for basic necessities members were on their own. With the passing of the communal kitchens, food was no longer free. Neither was shelter, and in exchange for stock, members could now become owners of the houses they lived in. Similarly, cobblers, tailors, and the like were urged to buy their shops, and set up as independent businessmen. For those who worked in the fields and mills, time clocks and pay checks were introduced....

"The effect was outstanding. Doubly spurred, because they were now both stockholders and wage earners, members fell to working harder.... Within a few months productivity had so risen that 200 outside workers could be discharged....

"From approximately $400,000 in 1932, total sales jumped to $727,000 in 1933. By 1942 they were up to $1,664,500; by 1948 to $7,058,500. The recession notwithstanding, business has continued as brisk as ever.... All in all, the society estimates, sales should reach $7,000,000. Being good capitalist businessmen, they wouldn't mind doing even better next year."

ROLE OF CONSERVATION OF RESOURCES

One of the most important factors in the development of the American economy to heights never before dreamed of has been the extensive basic resources with which the country was endowed. This economy did not develop in an exhausted country. The abundance of natural resources provides the basis for an abundant economy.

Our country has a high spiritual destiny—but a destiny that can only be realized if we guard these invaluable basic resources that make for the abundant economy. It is axiomatic, therefore, that our wealth of natural resources should not be used injudiciously, but rather the public and posterity have a right to expect their development to the best interests of the country, in so far as this can be done within the economic limitations prevailing.

In a talk before the Isaac Walton League at its last annual meeting, Dr. J. Alfred Hall presented a timely and effective commentary on the subject of conservation.

He points out that the system of free economic enterprise that we cherish, and is the most important part of our life, has within it the most important dangers to our way of life. Says Dr. Hall:

"Let's examine them a minute. Understand that under a system of unregulated free enterprise we have to do a few things. We have to mine the coal first that is closest to the top of the ground and of the best quality. Up in Minnesota we had to take out the best iron ore first. We had to cut the trees that were easiest to get out along Puget Sound. The biggest trees. We got rid of them first. We cleared the best agricultural ground. Then we wasted an awful lot of that. All the way through our whole fabric our system compels us to take the best first, and unfortunately it has so far compelled us to liquidate it rather rapidly. Now, the base of our life, as I see it, is an abundance of easily available rich natural resources, and this concomitant of our freedom, a system of free economic enterprise, tends to destroy that base. It can only live upon that base, but it tends to destroy it.

"I think the medical profession has

the term 'self-limiting disease.' This might be self-limiting, if we continue along the road that we have followed so far.

"All right, what do we do about it? There are two or three ways of going at it. One is the authoritarian way which none of us would want. Oh, sure, pass a a law! Pass a law! I hear that so many times. You cannot do anything with law in this country unless it is supported by the main body of public opinion. No law can get anywhere unless we support it because we are the government and what we say goes. So unless you have a kind of government that you and I don't want, even to think about it is not going to amount to anything.

"What is the alternative? Let her go. Well, you know what the alternative to let her go is. We have been letting her go for three hundred years. High, wide, and handsome. Come and get her. Pie in the sky! Free for everybody! That has been our philosophy and it has been a grand picnic for a lot of folks. But as you look down over the horizon a little you don't have to be an all-wise man to see that those things come to an end.

"What are we doing? We are getting ready to work lower grade iron ore. We are working lower grade aluminum. We are going farther and farther afield to get our coal. There is still a lot of coal, thank heaven. We are not going to run out of it. You know the forest situation as well as I do. A lot of these basic resources—copper—well, you know the copper story. We are already short a lot of things, and if we fight another war—a big one this one will be—we will pour out a tremendous lot of treasures, a tremendous lot of our resources. We will come out of it military victors but we will lose a lot of our resources base, and as we lose our resources base we lose a part of our liberty just as surely as if we were invaded and conquered by an alien enemy.

"Get that into our heads and I think we can lick this thing. We lose our liberty as we lose our stuff. Communism does not develop in countries like ours. Communism develops in countries where people have nothing to hope for.

"In the meantime, we have our job to do. So there is only one alternative that I see. That is an awakened moral conscience on the part of the people. That will give form and direction to this positive philosophy of the conservation of the base of our way of life."

No doubt most Americans agree with Dr. Hall that control by regulation and legislation should be kept to a minimum. It is abuses that often prompt legislation enacted in the public interest to protect our natural resources. Familiar examples are those relating to stream pollution, which tends to become more important with increasing population and increasing industrial development, to fisheries and to game.

Our challenge is the acceptance of the obligation of the conservation of resources by wise use, and the exercise of what may be called "self control" in the public interest, or "an awakened moral conscience on the part of the people."

Conservation and the ASTM:

We do not think of the ASTM primarily as a conservation organization, yet in its broader sense it is working extensively in this field. The establishment of standard methods of test for the evaluation of the properties of materials is an important factor in their efficient use, and the obtaining of reliable data also facilitates the making of substitutions when necessary. The many ASTM specifications for materials and products establish maintenance of quality or performance, at a known level through proper control. These materials and products can accordingly be used more efficiently and economically, and more definite or lower factors of safety can be employed. Through the ASTM procedure, these specifications are adopted voluntarily and serve in the public interest. They contribute materially to better economical utilization of our material resources, and afford in a concrete way, an illustration of the significant role of the ASTM in conservation.

ROLE OF STANDARDIZATION AND MATERIALS SPECIFICATIONS

Another important factor in American industrial development inviting special comment is the role of standardization and material specifications.

Standardization, particularly as it relates to quality and mass production, relatively speaking is a modern idea. In the handicraft era, it was unknown, or at most was no more than a dream.

Seventeenth Century Standardization:

Because of the premium for superior achievement, it is not surprising to find that thoughts of standardization were expressed at least as far back as three centuries ago, during the romantic days of iron-clad knights and their trusty steeds. The situation existing at that time led to the appointment of a commission by Charles I, that reported in 1631 on the need of standardizing armor, guns, pikes, and bandaliers, as follows:

> "And because we are credibly given to understand that the often and continual altering and changing of the fashion of armes and armours, some countrys and parts of the Kingdome having armours of one fashion, and some of another, do put many of our subjects to a great and unnecessary charge, and more than need requireth for the avoiding whereof, our will and pleasure is, and we do hereby appoint and command, that hereafter there shall be but one uniform fashion of armours of the same common and trayned bands throughout our said Kingdome of England and domynion of Wales, when as any of the said armours shall be supplied and new made, and that form and fashion of armour shall be agreeable to the last and modern fashion lately set down and appoynted to be used by the lords and others of our Council of Warre (the patterns, whereof are now and shall remayn in the office of our ordinance from tyme to tyme, which is our pleasure likewise concerning gunnes, pikes, and bandaliers whereof patterns are and shall remayn from tyme to tyme in our said office)."

Early Developments of Interchangeability of Parts:

However, it was not until 1801 that the first real example of the interchangeability of parts was demonstrated by the versatile Eli Whitney. In the book, "Masters of Mass Production," it is reported that he had accepted an order of 10,000 muskets from the War Department on March 1, 1799, with a time limit of 2 years for completion. At the end of the first year he had completed only 500 guns. He had become so engrossed in developing methods of production that his order was far from filled when the contract expired. Something needed to be done, so Eli Whitney brought a box to the War Department in Washington that he said held 10 muskets. When opened, it held no muskets, but a jumble of barrels, stocks, triggers, locks, and other parts. He asked an ordance expert to pick out a set of parts and lay them together. Quickly he converted the pile into a finished gun and continued the process until there were 10 guns. Nothing like that had ever happened on this planet before.

Need for standard specifications for quality and uniformity of product, and the development of methods of test, has come with the era of the industrial revolution. It is in these latter fields that the ASTM has played an all-important part.

METHODS OF ESTABLISHING STANDARDS AND SPECIFICATIONS

There are obviously a number of ways in which standards and specifications may be established—by government, by producers, and by voluntary multilateral agreement among producers, consumers, and general interests. It is by the voluntary action method that agencies such as the ASTM operate.

It has recently been pointed out by Mr. Lowell B. Mason of the Federal Trade Commission (*Standardization*, January, 1951), that there has been some fear that the adoption of standards, and presumably also of specifications, by industry groups may be regarded in some economic circles as "Conscious Parallel Action" that should be prohibited.

To emphasize the complications and disadvantages of not having standards, he describes an imaginative place, not far from the shores of America where it can be easily visited, that he calls "Non Parallelia." It is a show place, where one can go and observe the limitations of its strange obsession—for in Non Parallelia all standardization is barred. Says Mr. Mason:

> "In Non Parallelia there would be no organized efforts devoted to the educational and scientific aspects of the standardization of measurements, materials, products, methods, operations, and nomenclature, in industrial production and distribution.
>
> "In Non Parallelia, if a man wanted to make bolts and nuts with threads $\frac{37}{91}$ of an inch wide, he would not have huge and powerful consumer groups pressuring him to make $\frac{3}{16}$-in. bolts instead. In fact, in Non Parallelia we would require all men to make their products just a little different in size from everybody else.
>
> "This idea that railroad tracks, as well as thousands of other items in constant and general daily use, should be the same width is a vicious product of the American way of life and leads to what, in some Government economic circles, is known as 'Conscious Parallel Action.'
>
> "In Non Parallelia there will be no dull repetitive existence such as the American householder and his wife now have.
>
> "Take for instance the insignificant, but quite important operation known as watering the grass. Here in America, every man's hose coupling is the same size.
>
> "Walk up and down the residential streets of our cities; knock on every door; and you will find the same drab similarity in lawn faucets.
>
> "There is nothing in American lawn tending that sets one man apart from his less successful neighbors.
>
> "When it comes to watering the grass, we have no men of distinction.
>
> "Non Parallelia will not use the interchangeable thread system developed under the auspices of the American

Standards Association in conjunction with the British and Canadians.

"Think what a boon it would be to live in a country where you definitely know your neighbor will not come over to your house to borrow your hose because it wouldn't fit anyway. Probably your hose will not even fit your own faucet. But that will only make watering the lawn a thrilling adventure rather than a routine.

"In Non Parallelia we are going to have all industrial problems worked out by Government bureaucrats who do not know anything about Dimensional Coordination, Combined Specifications for Materials and Installation Work, Tensile Strength of Hydraulic Cement Mortars, Identification of Piping Systems, Ductility of Bituminous Materials, Sieve Analysis of Mineral Filler, Weather Resistant Saturants, Cupola Malleable Iron Specifications, and the Photographic Exposure Computer.

"Because we bureaucrats do not know anything about these things we will be able to bring a fresh outlook to the world.... We would not allow the Modular principle in building construction; we reject the engineering approach and substitute the political approach. Mandatory construction details will be the order of the day instead of performance requirements. This will effectively check any encouragement of production of improved materials and processes.

"You may not want to live in Non Parallelia. Frankly, I wouldn't care to either."

NON UTOPIA

We all know that in America today specifications for materials and products are indispensable, and we recognize the important part the ASTM plays in their development. Have you ever considered what an industrial nation would be like without them?

Let us consider for a moment we are in an imaginary country that has no specifications. This country would be called Non Utopia and logically would be located near Non Parallelia.

Since there are no specifications, each industry is free to operate independently, with no responsibility to consumers or the public. Its joint mottos, one learns, are "*Laissez nous faire seules*" and "*Caveat emptor.*" ("Let us act for ourselves" and "Let the buyer beware.")

Such things as dimensions and sizes are standardized, but without specifications there are obviously no standards of quality. Various products are hence made and sold without regard to grading or quality standards. Naturally a wide range in performance of products and materials may be expected. But that is not the producer's concern, for after all is not the buyer responsible for what he purchases, is not quality mainly for the consumer's convenience, assurance, and benefit?

In Non Utopia the simple matter of watering the lawn at times becomes a gay experience. With perfect size standardization, your neighbor's hose is, of course, sure to fit your faucet installation. However, after attaching your borrowed hose and sprinkler, you find your neighbor has been a little careless in his purchase. Immediately after turning on the water, you discover a sizable leak in the hose. You don't mind being sprayed, because after all, are you not watering the lawn as well? You, of course, find it easy to repair the hose with tape, after which you again turn on the water Much to your surprise the increased pressure causes another leak. And so the gay adventure continues.

Non Utopia is relatively new, but misgivings are already being heard as to its future. Illustrative of these misgivings are reports that railroad accidents have reached alarming proportions due to faulty materials; that the airlines have been forced to suspend operations pending inspection of the planes; that serviceability of some products is extremely low, with increasingly high replacement costs; and that homes built as lifetime investments are deteriorating in unpredictably short periods before installment payments are completed. Conditions have reached such a state that remedial measures must be taken. It is learned that a mission has been sent to America to study its specification system, including methods of preparation.

The cartoon is an effective device to highlight by exaggeration some particular point. This word cartoon of exaggeration of Non Utopia, of course, has no part in fact. To paraphrase the movies, "The presentation is purely fictional, and no resemblance to any individual or company living or dead is implied or intended."

What is intended, however, is to emphasize by contrast the method of self-control in the free enterprise system, and particularly what constitutes the "Keystone" of the ASTM way, namely, its formula for technical committee structure in the development of standard specifications and methods of test, with a balance of all interested parties, producers, consumers, and general interests. What success the ASTM has had in the general acceptance of its methods and specifications stems from this sound concept of providing a medium for give and take among all parties concerned. To an even greater extent, it is the solid foundation for its expanding future development.

THE ASTM CHALLENGE OF THE FUTURE

It has been previously mentioned, in connection with our national economy, that "What our forefathers have bequeathed to us, we must earn to possess." As I see it, this thought is equally applicable to the ASTM. It means that achievement must not be taken for granted; that there is never time to rest on our oars; that we must continue to be alert in keeping up with advances in technological progress; that we must explore new fields of endeavor whenever their need is indicated; that we must actively continue research, one aspect of which is that necessary sifting and winnowing by which alone the truth can be found; that we must continue to survey our committee structure, without fear of discontinuing some line of endeavor that has been outmoded; that we must protect the formula for technical committee structure through the full representation and required balance of all interested parties, producers, consumers, and general interests; and that we must continue to grow.

These observations are self-evident; I do not need to add that we must continue to work, because I know the ASTM will continue to do that. I believe it is one of the most, if not the most industrious and productive technical associations in the country. I know you will keep it so.

Those of you who enjoy the vigor of outdoor life and reflect on the rigors of pioneering in a rugged land, are no doubt familiar with Robert Service's vivid description of the Gold Rush Country in his "The Spell of the Yukon." Do you recall how it goes:

"I wanted the gold, and I sought it;
I scrabbled and mucked like a slave.
Was it famine or scurvy—I fought it;
I hurled my youth into a grave.
I wanted the gold, and I got it—
Came out with a fortune last fall, —
Yet somehow life's not what I thought it,
And somehow the gold isn't all.
"No! There's the land. (Have you seen it?)
It's the cussedest land that I know,
From the big, dizzy mountains that screen it
To the deep, deathlike valleys below.
Some say God was tired when He made it;
Some say it's a fine land to shun;
Maybe; but there's some as would trade it
For no land on earth—and I'm one."

With apologies to Robert Service, someone has paraphrased a conception of the spirit of the ASTM:

"Yes, there's the ASTM, have you seen it?
It's the busiest group that I know,
From the officers and staff that serve it,
To the active committees below.
We salute those whose effort has made it,
Who knew work was no thing to shun,
And I'm sure there's many would trade it,
For no group on earth, and I'm one."

IN CONCLUSION

In conclusion, with this annual meeting, I must relinquish the high office with which you have honored me and retire to the status of past-president. I have appreciated and enjoyed being president of an organization that is so important to the well-being of our nation, but presidents must step aside when the time comes, and make way for new men and new ideas. I do not expect to "fade away," for the ASTM has a way of keeping past-presidents busy. I expect to keep my technical committee activities, and hope to continue the fine association this work has brought about.

I realize that my big moment has passed. I am comforted, however, by Arthur Guiterman's lines on "The Vanity of Earthly Greatness":

"The tusks that clashed in mighty brawls
Of mastodons are billiard balls.
The sword of Charlemagne the Just
Is ferric oxide—known as rust.
The grizzly bear, whose potent hug
Was feared by all, is now a rug.
Great Caesar's bust is on my shelf,
And I don't feel so well myself.

ANNUAL REPORT OF THE BOARD OF DIRECTORS

The Board of Directors presents to the members this annual report of its stewardship of the Society's affairs at the close of a year that marked steady progress in ASTM activities. The report records briefly the principal happenings and developments of the Society year, including actions by the Board on matters of policy and administration. A minor amendment of the By-laws is proposed. The growth of technical activities has been noted and reference made to district and national meetings. Publications, membership, finances, and cooperative activities are covered. At best, the report is but an outline of progress during the year; the full picture appears with the wealth of data, tests, and specifications for this annual meeting and in the publications issued during the year and others in process.

In its report a year ago, the Board took advantage of the opportunity afforded by the midway point of the century to remark briefly upon the growth of the Society from small beginnings at the turn of the century to its present position. Reference was made to the considerable expansion of technical activities since the recent war, and to the strong administrative and financial foundations upon which to build for the future. It was noted that the Society has established for itself an important place in the technical and economic life of the nation and is well prepared to carry on the "promotion of knowledge of the materials of engineering, and the standardization of specifications and the methods of testing" in the dynamic age in which we are living.

As we face into the second half of the twentieth century, the immediate problem confronting the Society is that created by the National Emergency that has been declared. In considering how the Society can best serve the nation in its herculean preparations for defense in a troubled and unsettled world, the Board as an immediate step offered the services of the Society and its committees to the Government, including especially the Armed Services and those agencies dealing with the problems of defense production, standards and specifications, and conservation of critical materials. The responses to this offer have been prompt and cordial, and several conferences have already been held with certain agencies to explore various channels of cooperation in both standardization and research. Included in these are contacts with various groups in the Department of Defense, including Ordnance Department of the Army, Bureau of Ships, U. S. Navy, Munitions Board Standards Agency, and the Research and Development Board; the Defense Production Administration through its Conservation Division, and the National Production Authority; and the General Services Administration, including especially the Federal Specifications Board.

Recalling the experience of World War II, the extreme importance of conservation, and the contributions made by the Society in those days through emergency revision of ASTM specifications and tests, the Board as a second step has reinstituted a procedure for such emergency revisions which is described in the next section of this report. This clears the way for prompt action by the Society through its technical committees where emergency revisions of ASTM standards will aid the steps being taken for conservation of critical materials and facilitate the procurement of materials to ASTM standards during the emergency. In addition, several of our technical committees are at work on matters having value to certain agencies, in one instance at the direct request of an Industry Advisory Committee of the National Production Authority.

The Board will continue to follow closely those developments incident to the emergency that affect the activities of the Society and knows that it can count upon the whole-hearted cooperation of the members and committees of the Society in making the maximum possible contribution to the nation and to industry at this time.

Another less somber prospect that lies ahead is the occasion of the Fiftieth Anniversary of the incorporation of the Society in 1902, which will be celebrated appropriately during the annual meeting next year to be held in New York City, June 23–27, 1952. Many plans are being made for a notable meeting, which as they develop will be announced to the membership in the ASTM BULLETIN.

Technical Activities

The extent of the technical committee activities in the Society during the year now closing will be evident from the committee reports to be presented at this annual meeting, covering new and revised standards and the results of researches, and from the record of actions during the year by the Administrative Committee on Standards. This committee is responsible for following the standardization activities of the Society and its annual report to the Board (see Appendix II) covers some interesting matters. An important function of the committee is to study desirable expansion of our standardization activities and important developments in this direction have stemmed from the committee's recommendations.

The Administrative Committee on Research, whose annual report to the Board appears as Appendix III, keeps in touch with the research activities of the committees and lends advice and assistance where desired. The report refers to several important current and contemplated researches, and records progress during the year in the collation of formal statements of many unsolved problems prepared by the committees for suitable publication.

The appended reports of the Administrative Committee on Ultimate Consumer Goods (Appendix V) and Administrative Committee on Simulated Service Testing (Appendix VI) cover briefly the matters upon which these committees have been engaged.

New Activities:

The new Technical Committee D-22 on Methods of Atmospheric Sampling and Analysis was organized at Society headquarters on January 9, 1951, with the following scope:

> The formulation of methods of atmospheric sampling and analysis, the selection of acceptable nomenclature and definitions, and the stimulation of research to accomplish the foregoing purposes.

L. C. McCabe has been elected chairman, F. S. Mallette, vice-chairman, and H. H. Schrenk, secretary. The Board's decision to appoint this committee was announced in the report a year ago, although the title of the committee then suggested, namely, "Sampling and Analysis for Atmospheric Pollution" has been modified to that given above.

In reporting the organization of this important new committee, the Board wishes to emphasize that the Society through the activities of this committee will limit its work in this field specifically to the matters covered in the above-quoted scope and will not concern itself with the problems of specifying limitations of pollution nor with other matters concerning biological and health problems.

As reported a year ago, the Board has authorized organization of a new Technical Committee E-10 on Radioactive Isotopes. The organization meeting of this committee under the chairmanship *pro tem* of George G. Manov will be held during the 1951 annual meeting.

Following the organization in February, 1950, of the new Committee E-13 on Absorption Spectroscopy, as mentioned in the report a year ago, the following statement of scope has been approved for this committee:

> The coordination and formulation of methods of spectroscopic analysis by absorption, fluorescence, and scattering techniques.

During the year the Board has considered recommendations from several sources that the Society undertake standardization work in the field of cellulose and cellulose derivatives. This is a tremendously broad field in which certain committees of the Society are engaged to a greater or less degree, such as the committees working in the fields of paint, paper, electrical insulating materials, textiles, adhesives, and plastics. In addition, other technical organizations, especially the American Chemical Society and the Technical Association of the Pulp and Paper Industry, are engaged in technical work on these materials. In order to bring all pertinent information together and to consider how the Society might best engage in standardization work in this general field, a conference of interested groups was held at Society headquarters last November under the auspices of the Administrative Committee on Standards.

This conference developed that there is a real need for standardization of test methods and specifications for Cellulose and Cellulose Derivatives, which contemplates the chemical uses of cellulose. Representatives from various ASTM committees participating in the conference favored the organization of a new committee, which by concentrating on the methods of test in this field could be of considerable help to the present committees. Representatives of the American Chemical Society and the Technical Association of the Pulp and Paper Industry pointed out that since standardization work by these societies is a secondary part of their work, they believed the formation of a new ASTM committee devoting its main attention to the field of standardization would be of great benefit to the producers and users of these cellulosic materials. Accordingly, the Board of Directors has authorized the formation of a new Technical Committee D-23 on Cellulose and Cellulose Derivatives and has authorized the President and Executive Secretary to proceed with its organization. As a first step, Forrest Simmons has been appointed chairman *pro tem* and W. W. Becker secretary *pro tem*. A steering committee has been appointed to lay the plans for organization of this work.

The subject of titanium metal and alloys thereof has come very rapidly to the fore in the past year and its importance has been greatly enhanced by the present national emergency. The Board has concurred in a request of Committee B-2 on Non-Ferrous Metals and Alloys that work on titanium and its alloys be assigned to that committee. Suitable additions to the personnel of the committee have been made and a group has been organized to work on titanium under the direction of Subcommittee

VIII on Miscellaneous Refined Metals and Alloys of B-2. Committee E-3 on Chemical Analysis of Metals is undertaking the development of methods of chemical analysis of titanium.

Committee Scopes and Jurisdictions:

The following actions of the Board regarding committee scopes and jurisdictions are recorded:

The scope of Committee D-10 on Shipping Containers has been expanded to permit the committee to extend its activities into the field of packaging. The following revised statement of scope has been approved:

> Nomenclature, definition of terms, test methods, performance specifications, and study of the effect of various factors influencing strength and serviceability relating to packaging, including packages, shipping containers, and pallets.

By arrangement between Committees E-2 on Emission Spectroscopy and E-3 on Chemical Analysis of Metals, the former committee has assumed responsibility for preparing and developing methods of sampling for purpose of spectrochemical analysis. Accordingly, the scope of Committee E-2 has been changed to read as follows by the addition of the italicized words:

> The formulation of methods of spectroscopic analysis by emission techniques, *including methods of sampling.*

Agreement has been reached between Committees A-1 on Steel and E-7 on Non-Destructive Testing regarding the writing of methods of non-destructive tests for steel products. It has been agreed in principle that Committee E-7 will write general procedures for the various non-destructive tests and that a product committee such as that on steel will use such procedures in developing tests and specifications for specific applications to products. In order to facilitate cooperation between the two committees and to make more effectively available the advice that E-7 can give to A-1 on the techniques of non-destructive testing, two additional representatives from Committee A-1 are being appointed on Committee E-7.

ASTM Exposure Test Sites:

The Board has adopted Rules Governing the Use of ASTM Exposure Test Sites by Other Than ASTM Committees. These are based upon recommendations of the Advisory Committee on Corrosion and will be published as an appendix to the Regulations Governing Technical Committees in the 1951 Year Book. Two applications from outside groups for use of one or more of the ASTM exposure test sites have been approved, one being the American Electroplaters Society and the other the American Welding Society.

Reinstitution of ASTM Emergency Procedure:

In view of the growing restrictions in the use of critical materials occasioned by the national emergency, and with the knowledge that certain committees of the Society are already faced with the necessity of emergency revision of ASTM specifications if the specifications are to continue to be used during the emergency, the Board of Directors has concluded that it is necessary to reinstitute a procedure for emergency alternate provisions and emergency standards such as was in effect during World War II. Accordingly the following procedure has been established and is now in effect:

PROCEDURE FOR EMERGENCY ALTERNATE PROVISIONS AND EMERGENCY STANDARDS

Adopted by Board of Directors, May 8, 1951

I. EMERGENCY ALTERNATE PROVISIONS

1. Emergency Alternate Provisions in ASTM specifications are issued by the Society in the interest of expediting procurement or conservation of materials during the period of national emergency. They are intended for use where they may be considered by the purchaser of the material as a permissible alternate for the specific application or use desired.

2. An emergency alternate provision shall first have the approval by letter ballot of the appropriate subcommittee of the sponsoring ASTM technical committee. Such approval shall require a two-thirds affirmative vote of the combined number of consumers and general interests voting and a two-thirds affirmative vote of the producers voting, and the results of the ballot shall be classified as to affirmative and negative votes of producers, consumers, and general interests. Secondly, the emergency alternate provision shall then have the endorsement of the chairman of the main committee, or a two-thirds affirmative vote of the main committee by letter ballot in accordance with the provisions of the Regulations Governing Technical Committees, Section 14 (a).

3. When these requirements have been met, the emergency alternate provision shall then be submitted to the Administrative Committee on Standards together with a covering report that (1) describes adequately the conditions which in the opinion of the committee require the adoption of the proposed emergency provision, and (2) gives the reasons for any negative votes that have been cast. If approved by the Administrative Committee on Standards, the emergency alternate provision shall be published with the specification affected either in the form of a sticker or as an accompanying sheet and shall also be published in the next succeeding issue of the ASTM BULLETIN. Any emergency alternate provisions approved during the year shall be recorded in the next annual report of the technical committee.

4. All such provisions are subject to annual review, and for them to be continued in effect they shall be reaffirmed by letter ballot of the Main Committee in accordance with the provisions of the Regulations Governing Technical Committees, Section 14 (a). The technical committee shall report its recommendation with respect to continuance or otherwise of emergency alternate provisions to the Administrative Committee on Standards.

5. Emergency alternate provisions will be identified by the letters "EA" preceding the serial designation of the specification with which they are issued. The date of issue will be given. If the emergency alternate provision is subsequently revised, lower case letters "a," "b," etc., will be added to the designation and a new date of issue shown.

Example: EA - B 32a

Issued January 1, 1952
(Superseding Issue of October 15, 1951)

There will also be printed with each emergency alternate provision the following note:

> These Emergency Alternate Provisions are issued by the American Society for Testing Materials in accordance with a special procedure in the interest of expediting procurement or conservation of materials during the period of national emergency. They are intended for use where they may be considered by the purchaser of the material as a permissible alternate for the specific application or use desired.

Emergency alternate provisions will be printed on pink paper.

II. EMERGENCY STANDARDS

6. Emergency Standards are likewise issued by the Society in the interest of expediting procurement or conservation of materials during the period of national emergency. They may be issued whenever a technical committee finds it appropriate to issue complete new emergency specifications or methods of test rather than emergency alternate provisions. They may be alternates to existing specifications or methods, in which case the existing specifications or methods will be suitably marked to indicate the existence of an alternate.

7. The procedures to be followed by the technical committee and the Administrative Committee on Standards in the development and promulgation of such emergency standards shall be the same as those specified for the approval of emergency alternate provisions in Sections 2, 3, and 4 above.

8. Emergency standards will be given an ASTM designation consisting of the letters "ES" followed by a number, beginning with 1 for the first ES, 2 for the second, etc. The date of issue will be given. If the emergency standard is subsequently revised, lower case letters "a," "b," etc., will be added to the designation and a new date of issue shown.

Example: ASTM Designation: ES 21b

Issued July 1, 1952
(Superseding Issue of January 1, 1952)

There will also be printed with each emergency standard the following note:

> Emergency Specifications (Methods) are issued by the American Society for Testing Materials in accordance with a special procedure in the interest of expediting procurement or conservation of materials during the period of National Emergency. They are intended for use where they may be considered by the purchaser of the material as suitable for the specific application or use desired, or as a permissible alternate for existing specifications (methods).

Emergency standards will be printed on yellow paper.

This procedure is similar to that in effect during the recent war and, as then, vests final authority for the issuance of emergency alternate provisions and emergency standards in the Administrative Committee on Standards, which will carefully examine all proposed emergency actions and make certain that all requirements of the emergency procedure are met.

The most important change in the procedure has been to tighten requirements for emergency action in the technical committees. In the earlier emergency procedure it was possible for a sub-group of a subcommittee to agree upon an emergency provision or standard without reference to the subcommittee or the main committee, requiring only endorsement of the chairman of the main committee. Under this procedure there was a tendency for such subgroups, which were sometimes not fully representative of all parties at interest, to rush things through under pressure and perhaps with not sufficient consideration of all sides of the question. Accordingly in the procedure now effective no provision is made for sub-group action; instead it is required that a two-thirds letter ballot approval of the appropriate subcommittee be obtained and also that the ballot be classified as to affirmative and negative votes of producers, consumers and general interests. As before, approval of the chairman of the main committee is required, or if desired a two-thirds affirmative letter ballot vote of the main committee. In this way protection of the interests of all parties will be obtained. In arranging for annual review of all emergency alternate provisions and emergency standards by the main committee, it is now required that for such emergency actions to be continued in effect they must be reaffirmed by a letter ballot of the main committee.

The Board feels that it is incumbent upon the committees of the Society to be certain that there is a real need for the issuance of emergency alternate provisions or emergency standards before recommendations pertaining thereto are submitted to the Administrative Committee on Standards, inasmuch as the promulgation of these emergency actions is both time consuming and expensive and should be done only where it is clearly necessary in the interest of furthering procurement of materials to ASTM standards during the national emergency.

In the preparation of this emergency procedure due account has been taken of the procedures recently adopted by the Federal Specifications Board and the Department of Defense. The principal difference is that the two government agencies have eliminated "emergency alternate provisions" and will issue mandatory conservation requirements as regular amendments. The FSB will indicate by a footnote where provisions of a regular amendment are intended to conserve critical or strategic materials, and when the emergency is over such amendments will be considered for revision. This practice is feasible when specifications are written by a consumer (in this instance the Government) primarily for its own use. However, ASTM specifications are written for use by hundreds of consumers. We cannot possibly foresee all the ramifications of use of materials under ASTM specifications and must therefore follow a procedure which leaves a choice to the consumer of using either the regular ASTM standard if he feels he must have the material covered therein and can obtain it, or using the emergency alternate provision if it will provide material suitable for his specific application. This thought has been carefully worked out in the note to be printed with each emergency alternate provision. Moreover emergency provisions may of necessity represent a temporary lowering of quality in some particular, in which case there would be serious objection to formulating a formal amendment which would remove the original requirement from the specification. Accordingly it is believed that the "emergency alternate provision" is an essential feature of our emergency procedure and is well adapted to our needs.

District and Related Activities

There were no major administrative matters requiring attention during the past year in the operation of the ASTM Districts. The thirteen districts that have been organized functioned satisfactorily under the current Charter and Manual, and most of the districts sponsored one or more meetings during the year.

In carrying out the primary purposes of districts, which are to advance the interests of the Society in a given locality and to further the purposes for which ASTM is organized, the districts frequently assist with national meetings and undertake other projects. The Philadelphia District acted as host for the 1950 Annual Meeting and is repeating in 1951, its third consecutive year. It has planned the Ladies' Entertainment, handled details of the Annual Dinner, and underwritten and sponsored the Entertainment Program. The New York District Council will cooperate closely with the Committee on Arrangements for the 1952 Annual Meeting in New York.

District Meetings:

The accompanying table lists all of the ASTM National and District Meetings during the year, with notes on the features of various district meetings. Several of these meetings were sponsored jointly with local sections of other organizations.

There was excellent attendance at most district meetings, and the programs were all interesting and pertinent.

President Markwardt was the technical speaker at several of the meetings, presenting in a very informative and interesting way data on forest products research and on lightweight composite constructions. His paper "Highlights on Progress in Forest Products Research" was published in the May 1951 BULLETIN and his paper on "Developments and Trends in Lightweight Composite Con-

ASTM MEETINGS, JUNE 1950–MAY 1951, INCLUSIVE.

Meeting	Place and Date	Notes
Annual Meeting	Atlantic City, N. J. Hotel Chalfonte-Haddon Hall June 26–30, 1950	23 Technical Sessions; Several Informal Round Table Discussions, and Groups of Papers 500 Committee Meetings (Registered Attendance 2131—408 Ladies)
ASTM Committee Week	Cincinnati, Ohio Netherland Plaza Hotel March 5–9, 1951	200 Committee Meetings (About 835 in attendance)
Spring Meeting	Cincinnati, Ohio Netherland Plaza Hotel March 7, 1951	Symposium on Thermal Insulating Materials[a] (About 250 in attendance)
District or Local (sponsored by Councils indicated)	New York, N. Y. Engineering Societies Bldg. October 6, 1950	Effects of Atomic Bombing, by Edward J. Kehoe[b]
	Columbus, Ohio (Ohio Valley District) Ohio State University October 13, 1950	Air Pollution, by H. G. Dyktor;[c] Water Pollution, by L. F. Warrick[c]
	Pittsburgh, Pa. University Club November 6, 1950	Smoker: Informal Remarks by President L. J. Markwardt and Executive Secretary C. L. Warwick
	Cambridge, Mass. (New England) Commander Hotel November 8, 1950	Highlights of Progress in Forest Products Research, by L. J. Markwardt[d]
	Cleveland, Ohio Case Institute of Technology November 14, 1950	The Search for Iron Ore, by D. B. Gillies[e]
	Chicago, Ill. Western Society of Engineers Headquarters, November 27, 1950	Jointly with Western Society of Engineers Highlights of Progress in Forest Products Research, by L. J. Markwardt[d]
	Buffalo, N. Y. (Western New York - Ontario) Hotel Sheraton November 28, 1950	Highlights of Progress in Forest Products Research, by L. J. Markwardt[d]
	Los Angeles, Calif. (Southern California) Southern California Edison Co. November 30, 1950	Application of New Material to Telephone Communications, by A. P. Hill
	Philadelphia, Pa. Franklin Institute November 30, 1950	Our Forests and Streams—Priceless Economic and Industrial Assets—Are We Conserving Them?, by L. A. Danse
	New York, N. Y. Engineering Societies Bldg. January 12, 1951	Jointly with Machine Design Division, A.S.M.E. Titanium: Derivation and Mill Fabrication, by T. W. Lippert; Alloys—Their Potential Applications, by N. E. Promisel
	Philadelphia, Pa. Franklin Institute January 16, 1951	Development and Trends in Light-weight Composite Construction, by L. J. Markwardt[f]
	Cleveland, Ohio Hotel Cleveland February 5, 1951	Development and Trends in Light-weight Composite Construction, by L. J. Markwardt[f]
	St. Louis, Mo. Engineers Club February 20, 1951	Highlights of Progress in Forest Products Research, by L. J. Markwardt[d]
	Philadelphia, Pa. Franklin Institute February 20, 1951	Metallurgical Development, by W. A. Reich[g]
	Portland, Oregon (Local Group) Burns Restaurant March 8, 1951	Jointly with Other Cooperating Societies Highlights of Progress in Forest Products Research, by L. J. Markwardt;[d] Highlights of Current ASTM Work in Materials, by C. L. Warwick
	San Francisco, Calif. (Northern California) Engineers Club March 13, 1951	Jointly with Structural Engineers Assn. of Northern California Wood as an Engineering Material, by L. J. Markwardt; Highlights of Current ASTM Work in Materials, by C. L. Warwick

ASTM MEETINGS, JUNE 1950–MAY 1951, INCLUSIVE—(*Continued*)

Meeting	Place and Date	Notes
District or Local (sponsored by Councils indicated)	Los Angeles, Calif. (Southern California) Rodger Young Auditorium March 16, 1951	Highlights of Progress in Forest Products Research, by L. J. Markwardt;[d] Features of Current ASTM Work in Materials, by C. L. Warwick
	Philadelphia, Pa. Franklin Institute March 22, 1951	Symposium on Wear Testing: Wear of the Human Body, by J. P. Hubbard; Use of Radioactive Tracers in Automotive Petroleum Research, by F. C. Burk; Textiles, by C. L. Simon; Metals, by C. D. Strang, Jr.; Highways, by N. W. Carey, Jr.
	Pittsburgh, Pa. Mellon Institute March 29, 1951	Joint Meeting with Pittsburgh Section Instrument Society of America and Pittsburgh Section Society for Experimental Stress Analysis Stress Analysis in Action, by W. T. Bean, Jr.
	New York, N. Y. Engineering Societies Bldg. April 20, 1951	Meeting on New Synthetic Fibers—What Do They Mean to Us? Papers on: Synthetic Fibers, by C. W. Bendigo; Dacron Polyester Fiber (Formerly Fiber V) and Orlon Acrylic Fiber, by J. B. Quig; Dynel—Its Characteristics and Uses, by C. A. Setterstrom; Dyeing and Finishing Synthetic Fibers, by A. W. Etchells[h]
	St. Louis, Mo. Engineers Club April 24, 1951	Modern Photography in Industry, by R. M. Woodward
	Hartford, Conn. (New England) Hartford Canoe Club April 26, 1951	Corrosion, by H. H. Uhlig
	Detroit, Mich. Rackham Memorial Bldg. May 3, 1951	Remarks by President L. J. Markwardt; High-Temperature Metals, by A. E. White

[a] Issued as a separate publication, *STP No. 91*, January, 1952.
[b] Extended abstract published in ASTM BULLETIN No. 170, December, 1950, p. 16.
[c] Notes appeared in the ASTM BULLETIN No. 170, December, 1950, p. 13–14.
[d] Published in ASTM BULLETIN No. 174, May, 1951, p. 45-54.
[e] Abstracted in ASTM BULLETIN No. 171, January, 1951, p. 28.
[f] Published as part of the 1951 Symposium on Structural Sandwich Construction, *STP* No. 118, January, 1952, p. 3.
[g] Extended abstract published in ASTM BULLETIN No. 174, May 1951, p. 31–32.
[h] Abstracted in ASTM BULLETIN No. 175, July, 1951, p. 40.

struction" is to appear in the 1951 Symposium on Structural Sandwich Construction to be published later in the year.

At most of the district meetings, someone from the Headquarters' Staff was present, either the Executive Secretary or one of his associates.

First Meetings in Several Cities:

In October, 1950, during a trip in the Southeast, the Assistant Secretary arranged luncheon meetings of our members in Birmingham and Atlanta. This was the first time that our members had met in these two industrial centers. An ASTM meeting was held for the first time also in Hartford, Conn., on April 26, 1951, when the New England District sponsored a technical meeting. There were about 140 present.

Contacts with Members in the West and Southwest:

During the course of a trip to the West Coast by the President and Executive Secretary, district meetings were arranged in San Francisco and Los Angeles, which were preceded by a meeting in Portland, Ore. At each of these meetings, the President, as noted in the accompanying table, presented a technical paper, and the Executive Secretary spoke on features of current Society work.

On his return journey from Los Angeles, the Executive Secretary attended meetings which had been arranged in Dallas, Houston, and Birmingham. He spoke at each meeting, following which there was an informal discussion with the members present.

In each industrial area, the officers visited the offices and plants of many of our members and other leading industrial establishments.

It is quite evident that with the great industrial development in each of the localities visited, and especially in the Southwest, there are many opportunities for extension of the Society's membership. There are loyal and influential groups of ASTM members in these areas.

National Meetings

The Society's Fifty-Third Annual Meeting held in Atlantic City June 26 to 30, 1950, was marked by the highest registered attendance for any such meeting and by a very diversified and extensive technical program. There were about 500 meetings of various technical committees and their sub-groups. Also in progress throughout the week of the meeting was the Society's Ninth Exhibit of Testing Apparatus and Related Equipment, together with the Photographic Exhibit which featured sections on photomicrography and electron micrography. Total attendance of 2131 compares with the previous high of 2063 in New York in 1944. Over 400 ladies also attended the meeting. The Philadelphia District, which acted as host, provided an interesting entertainment program, in which many of the ladies participated.

The technical program was marked by several sessions dealing with the effect of temperature on the properties of materials. The Symposium on the Rôle of Non-Destructive Testing in the Economics of Production is considered outstanding, and was developed to acquaint management with the advantages and uses of non-destructive testing.

The 1951 Spring Meeting was held in Cincinnati on March 7 during ASTM Committee Week extending from March 5 to 9, inclusive. The Symposium on Thermal Insulating Materials, to be published in the summer of 1951, featured the Spring Meeting. The registration for Committee Week totaled 835. There were about 200 meetings of the Society's technical committees.

Reference should be made to a session jointly sponsored by ASTM with the Forest Products Research Society during the latter society's annual meeting held in Philadelphia May 7 to 11, 1951. The several papers at the session were sponsored by Committee D-7 on Wood; President Markwardt presided at the session.

1952 and 1953 Meetings:

The 1952 Annual Meeting will commemorate the Society's Fiftieth Anniversary, ASTM having been incorporated as a technical society in 1902. This meeting, together with the Tenth Exhibit of Testing Apparatus and Related Equipment and Biennial Photographic Exhibit will be held June 23 to 27 in New York City, with the Hotel Statler as headquarters.

The 1952 Committee Week and Spring Meeting will be held in Cleveland at the Hotel Statler, during the week of March 3.

The Society will participate in the Centennial of Engineering to be held in Chicago in September, 1952, commemorating the One-hundredth Anniversary of the founding of the American Society of Civil Engineers.

In 1953 the Society will return to Chalfonte-Haddon Hall, Atlantic City, for its Fifty-sixth Annual Meeting, June 22 to 26, inclusive.

The 1953 Committee Week and Spring Meeting will be held in Detroit, during the week of March 2.

Publications

Responsibility for the publications of the Society is delegated to the Administrative Committee on Papers and Publications. The report of that committee is attached as Appendix IV; it reviews the activities of the committee and gives interesting information about the publications for the past year as well as some future publications.

The annual volume of the *Proceedings* (1480 pages) and the six 1950 Supplements to the Book of Standards (approximately 2180 pages) were the major 1950 publications. The magnitude of these publications imposed a heavy task on both the editorial staff and the printing establishment, and the final book was not completed until May, 1951. Fifteen compilations of standards and related publications were printed and together with various special publications listed in the Committee's report comprise an important part of the 1950 publications.

Included in the 1950 publications, and contributing in no small measure to the editorial and publication "load" of the year, were the 53 papers from the Pacific Area National Meeting in San Francisco, October, 1949, that have now been published. Fifteen of these have appeared in the BULLETINS of 1950 and 1951; six are included in the 1950 *Proceedings*; and the remaining 32 have appeared as Symposiums in seven Special Technical Publications. These papers comprise 912 pages (6 by 9 in. format), approximately three-fourths the size of the average volume of *Proceedings*. It is gratifying to record the high quality of these papers, for they constitute a notable contribution to ASTM publications.

Excellent progress is now being made in the final editing and typesetting of the Manual of Oil Measurement Tables being compiled jointly by ASTM and the British Institution of Petroleum. It is hoped that the tables covering U. S. and British units will be available during 1951.

The Manual for Authors of ASTM Papers, mentioned in the report a year ago, has now been completed and constitutes an important aid to authors and to the editorial staff. Many individuals and organizations are requesting copies.

Administrative Matters

Amendment of By-laws:

In the light of experience under the procedure for election of Honorary Members in the present provisions of the By-laws, the Board recommends a modification whereby a proposal for honorary membership may be submitted to the Board by any member of the Society instead of by at least ten members. Accordingly, the Board recommends to the annual meeting that the following amendment to Article I, Section 7 of the By-laws—which in accordance with Article X of the By-laws on Amendments has been duly proposed and acted upon by the Board at its meeting on May 8, 1951—be passed to a letter ballot of the Society membership for adoption:

Delete the following sentence:

> A nominee for honorary membership shall be proposed to the Board of Directors by at least ten members.

Substitute the following sentence:

> Nominees for honorary membership may be proposed in writing to the Board of Directors by any member of the Society.

Microfilm of Certain Vital Records:

Microfilm copies of membership and committee membership records and of membership ledger cards as of October 15, 1950, and of all the Year Books of the Society, have been made and stored in a safe deposit box at the Bellefonte Trust Co., Bellefonte, Pa. This action was taken in order to have these vital records, which for the most part exist nowhere else, stored in a place presumably free from danger of destruction by bomb attack. These records provide a basis from which to reconstruct membership and other data that might be destroyed at Society headquarters in the event of a bomb attack on Philadelphia. Consideration is being given to periodic microfilming of such current membership and financial records as may seem advisable.

It has not been considered necessary, from the viewpoint of safety from bomb attack, to microfilm the technical publications of the Society, especially the Book of Standards and Proceedings, since complete sets of these publications are available in many places throughout the country, including most of the principal libraries.

Headquarters Staff:

In March, 1951, William W. Menz was engaged as an assistant editor, replacing William F. Black, Jr., who had resigned. The Board has created an additional position in the technical and administrative staff, together with the necessary supporting stenographic assistance. When these positions are filled the headquarters staff will total 57.

The following staff promotions were made during the year: G. A. Wilson to the position of Senior Assistant Editor; M. D. Huber, to the position of Assistant Standards Editor; and J. W. Caum, to the position of Assistant Technical Secretary.

On May 1, 1951, Miss Dorothy E. Hand, Chief of the Sales Section, Finance Department, completed twenty-five years of service as a member of the staff. Appropriate recognition of Miss Hand's services was made by the Board at its May meeting.

Society Headquarters:

The headquarters have continued to be regularly used by committees and members of the Society. In the calendar year 1950 there were 94 committee meetings and conferences held in the building, including several meetings of other organizations to whom the Society is always pleased to extend this courtesy.

Later in this report is given full information regarding the operation of the Building Fund through which the purchase of the headquarters was made. The headquarters building is carried on the Balance Sheet as an asset after depreciation of $166,340.87. The building is nearly clear of indebtedness, there being only a note payable to General Funds of $12,733.34 as of December 31, 1950. Provision is made in the budget under Headquarters Occupancy Expense to pay off this loan at the rate of approximately $1130 a year.

The cost of operating the headquarters building in 1950, including full depreciation charges and portions of staff salaries chargeable to management and maintenance services, was about $2 per sq. ft.

The Board has set aside the sum of $25,000 as a Headquarters Building Reserve to be applied if and as required to expand Society headquarters. The rapid growth of Society activities, and hence of its staff, has brought this problem of expanded headquarters to a head more rapidly than was anticipated, and the Board has begun a study of the matter.

Membership

On June 1, 1951, the membership of the Society totaled 7001, compared with a total of 6761 one year ago. Gains and losses in various classes of membership are shown in the following table:

Class of Membership	Membership		Losses				Additions		Total		
	June 1, 1950	June 1, 1951	Resignations	Dropped	Death	Transfer	Transfer	Election	Loss	Gain	Increase
Honorary	16	22	...	...	...	...	6	...	...	6	6
Perpetuity and Life	11	11	...	...	...	...	...	...	...	...	...
Sustaining	214	236	...	...	...	1	20	3	1	23	22
Company, Firm, etc.	1658	1704	29	29	...	25	9	120	83	129	46
Individual, etc.	4739	4918	203	86	41	15	23	501	345	524	179
Junior	123	110	8	16	...	17	...	28	41	28	13[a]
Total	6761	7001	240	131	41	58	58	652	470	710	240
Student	493	458	22	284	...	16	...	287	322	287	35[a]

[a] Net loss

It will be noted that 652 new members were elected, compared with 627 for the preceding year. The larger number of new members, coupled with the fewer losses from resignations, delinquencies and death, 412 this year compared with 486 the previous year, resulted in a net increase of 240, which compares with the net increase reported a year ago of 141. It will be seen from the table that there was a substantial increase in the number of sustaining, company, and individual memberships.

Considering that our membership is now just over 7000, it is interesting to compare this with the figures for ten and twenty years ago: in 1941 our total membership was 4470, and in 1931 it was 4327. A true comparison would, of course, need to consider the relative percentages of sustaining, company, and individual memberships. Nevertheless the steady growth in our total membership is encouraging.

Members in the Armed Services:

Some members of the Society have recently entered the Armed Services of our country. By action of the Board, all such members will be continued on the membership rolls of the Society with remission of dues, and will receive the ASTM BULLETIN mailed to any designated address.

Sustaining Members:

There have been twenty-three new sustaining members, and there was one loss, being a transfer to company membership. The new sustaining members are:

Alpha Portland Cement Co.
Armour and Co.
A. M. Byers Co.
Continental Steel Corp.
Deere and Co.
Deering, Milliken and Co., Inc.
Eastern Gas and Fuel Associates
General Portland Cement Co.
Great Northen Railway Co.
Hamilton Watch Co.
Heppenstall Co.
Industrial Rayon Corp.
Johnson & Johnson
Joy Manufacturing Co.
The M. W. Kellogg Co.
Minneapolis-Honeywell Regulator Co.
Pacific Gas and Electric Co.
Scovill Manufacturing Co.
Standard Steel Spring Co.
U. S. Industrial Chemicals, Inc.
United States Smelting, Refining and Mining Co.
Universal-Cyclops Steel Corp.
Weston Electrical Instrument Corp.

Several other companies have signified their intention of becoming sustaining members.

40-Year Members:

There is a total of 26 members who this year have completed forty years of membership, which brings the total number of certificates issued to the "Forty-Year Club" to 184. The members who will receive the 40-Year Certificates at this annual meeting are as follows:

American Locomotive Co.
Baldwin-Lima-Hamilton Corp.
Bridgeport Brass Co.
Brown & Sharpe Mfg. Co.
Carnegie Institute of Technology
General Motors Corp.
S. H. Graf
Hercules Cement Corp.
S. H. Ingberg
Ledoux and Co., Inc.
J. O. Leech
J. H. Nead
R. H. Noderer
Northern Electric Co., Ltd.
Monroe L. Patzig
Pratt & Lambert, Inc.
W. P. Putnam
Louis G. Robinson
The Steel Co. of Canada, Ltd.
United States Gypsum Co.
U. S. Department of the Navy, Bureau of Aeronautics
U. S. Department of the Navy, Bureau of Ordnance
U. S. Department of the Navy, Bureau of Ships
U. S. Naval Engineering Experiment Station
Herbert L. Whittemore
Lucien I. Yeomans

Deaths:

The Society has lost the following 41 members through death:

Ainsworth, Allan W. (1937)[a]
Anderson, A. G. (1943)
Bainbridge, William C. (1928)
Beall, Almon L. (1933)
Boekel, Julius (1923)
Bowman, John J. (1949)
Briggs, Clay (1936)
Coltman, Robert W. (1919)
Corman, H. E. (1947)
Crum, Roy W. (1911)
Dunglinson, Basil (1937)
Duval d'Adrian, A. L. (1937)
Finkbiner, N. M. (1925)
Fisher, Russell T. (1922)
Fitts, James Logan (1925)
Fleming, J. W. (1950)
Gaskill, P. D. (1925)
Goetz, Alvin C. (1939)
Harrington, L. C. (1932)
Heussner, Carl E. (1933)
Hughes, E. M. (1920)
Joos, Charles E. (1936)
Lukash, John G. (1944)
Lundell, G. E. F. (1918)
Macdonald, John B. (1924)
McElwee, R. G. (1936)
Moulton, M. V. (1935)
Mueser, William (1898)
Mulligan, Maurice J. (1948)
Randall, Frank A. (1921)
Rawson, R. H. (1949)
Robertson, H. H. (1912)
Roth, H. A. (1943)
Sandell, Bert E. (1937)
Scoular, Spencer Gray (1939)
Skinner, Charles Edward (1903)
Southard, John C. (1948)
Stark, Harry W. (1930)
Sweetser, Ernest O. (1927)
Zeder, Fred M. (1917)
Zeitfuchs, E. H. (1947)

In addition to the above, the following representatives of company members of the Society passed away:

F. E. Bash, Driver-Harris Co.
H. D. Baylor, Louisville Cement Co.
F. A. Bond, The McKay Co.
Alfred Ewing, Climax Rock Drill and Engineering Works
C. F. Lauenstein, Link-Belt Co.
Nathan Lester, Lester Engineering Co.
W. G. Hooper, Midwest Piping and Supply Co., Inc.
William McDowell, Electro Metallurgical Division, Union Carbide and Carbon Corp.
Guy H. Merry, Merry Brothers Brick and Tile Co.
Harry T. Newman, New York City Department of Purchase
David C. Scott, Scott Testers, Inc.
Howard M. Smith, Wyckoff Steel Co.

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[a] Date of Membership.

Frank Stutz, Better Fabrics Testing Bureau
J. Hall Taylor, Taylor Forge and Pipe Works
J. N. Wickert, Carbide and Carbon Chemicals Div., Union Carbide and Carbon Corp.
M. Allan Wilson, Delaware State Highway Dept.
M. C. Wylie, Gulick-Henderson Laboratories, Inc.

A number of these men have been affiliated with the Society for many years and rendered outstanding services, particularly through work on the technical committees. Dr. G. E. F. Lundell, for many years Head, Chemistry Division, National Bureau of Standards, was past-president of the Society, and to him must go much credit for the important accomplishments of the Society in the field of chemical analysis of metals and of other materials. Before Committee E-3 on Chemical Analysis of Metals was established, he served on many of the metals committees guiding their work in establishing analytical methods, and was chairman of Committee E-3 from 1935 to 1948. The past year has taken its toll among some of our older and, too, among relatively younger members. John Bowman, in the latter classification, had nevertheless rendered outstanding service in several capacities, notably, as Secretary of Committee B-7 on Light Metals and Alloys, as Secretary for ten years of Committee E-4 on Metallography, and as Chairman of the Pittsburgh District. Roy W. Crum, one of the older members, had an outstanding record of service, particularly in Committee C-9 on Concrete and Concrete Aggregates, of which he was a past-chairman and honorary member, but he also served on many other technical groups, including Committee D-4 on Road and Paving Materials of which he had been chairman. Russell T. Fisher had been active in the work of Committee D-13 on Textile Materials for many years. A. C. Goetz was especially active in Committee D-1 on Paint, Varnish, and Related Products, but his interest covered other committees as well. He served on the Ohio Valley District Council. Carl E. Heussner made many contributions to the Society's work, notably in the field of die-cast metals and alloys, corrosion of non-ferrous metals and alloys, and electrodeposited metallic coatings, serving on our committees in these fields. He served on the Society's Committee on Papers and Publications and was active in the Detroit District.

In the death of William Meusser the Society loses one of its three individual members who have been affiliated with the Society continuously since 1898.

Frank H. Randall was a loyal ASTM member and served on the Chicago District Council. Bert E. Sandell had served for many years on the ASTM committees on die castings and light metals and alloys. Charles Edward Skinner is another of the long-time members (1903) who particularly in the early years of the Society took a most constructive part in our work and maintained his interest in ASTM down through the years. He was the first chairman of Committee D-9 on Electrical Insulating Materials. He was active in the early International Association for Testing Materials and was a member of the ASTM Board of Directors in 1912.

F. E. Bash made many important contributions to our work, especially in Committee B-4 on Electrical Heating, Resistance, and Related Alloys. He had been secretary of this group virtually since its formation. He was an active member of other committees. H. D. Baylor was for many years a member of Committee C-1 on Cement and other groups and took a leading part in organizing Committee C-12 on Mortars for Unit Masonry.

David C. Scott, Sr., a member of

ASTM for many years and a pioneering manufacturer of testing equipment, was for many years active in Committee D-13 on Textile Materials and in Committee D-11 on Rubber Products. Howard M. Smith was concerned with the work of Committee A-1 on Steel. J. Hall Taylor served on Committee A-1 on Steel for many years and was chairman of its subgroup on forgings for high-temperature service for some time. Melville C. Wiley, actively interested in ASTM work for many years, had served on the Pittsburgh Council for several terms.

Honors and Awards

Periodically the Board of Directors, acting for the Society, takes cognizance of outstanding service through election to Honorary Membership, and the Award of Merit recently established by the Board provides a further means of recognizing important service to the Society. Various awards and medals are also given to recognize outstanding papers and to foster research in certain special fields of activity.

Honorary Membership:

During this annual meeting the following five members, whom the Board recently elected to Honorary Membership, will be presented to the Society for their formal awards; each has over long periods of years contributed greatly to the advancement of the Society's work:

Thomas G. Delbridge (retired), for many years with Atlantic Refining Co., and Past-President of the Society.

Albert T. Goldbeck, Engineering Director, National Crushed Stone Assn., Inc., and Past-Director of the Society.

Harold H. Morgan, Vice-President and Chief Engineer, Robert W. Hunt Co., and Past-President of the Society.

Frank E. Richart, Research Professor of Engineering Materials, University of Illinois, and retiring Vice-President of the Society.

Frederick W. Smither (retired), formerly Chemist, National Bureau of Standards.

Award of Merit:

The Award of Merit Committee for 1951, comprising L. H. Winkler, *chairman*, Dean Harvey, C. H. Rose, R. L. Templin, and L. J. Trostel, reviewed numerous suggestions and citations from the various technical committees and nominated ten members who by unanimous action of the Board were voted the 1951 Awards of Merit. Each one has contributed in an important way to the work of the Society in various of our technical fields. They will be presented to the Society during this annual meeting to receive the Award of Merit certificates:

William Blum
Hyman Bornstein
Robert Burns
H. V. Churchill
Max Hecht
Carl D. Hocker
William H. Klein
H. H. Lester
Stanton Walker
William H. Whitcomb

Recognition of Technical Papers:

In recognition of certain outstanding technical papers, four awards will be made at this annual meeting as follows:

Charles B. Dudley Medal—presented for a paper of outstanding merit constituting an original contribution on research in engineering materials. This year it will be presented to D. S. Clark and P. E. Duwez, of the California Institute of Technology, for their paper on "The Influence of Strain Rate on Some Tensile Properties of Steel."

Richard L. Templin Award—presented for a paper describing new testing methods and apparatus, the purpose of the award being to stimulate research in the development of testing methods and apparatus. This year it will be given to Richard L. Templin and W. C. Aber, of the Aluminum Co. of America, for their paper entitled "A Method for Making Tension Tests of Metals Using a Miniature Specimen."

Sam Tour Award—presented for the purpose of encouraging research on the improvements and evaluation of corrosion testing methods and to stimulate the preparation of technical papers in this field. This year the award will be made to C. T. Evans, Jr., Elliott Co., for his paper entitled "Oil Ash Corrosion of Materials at Elevated Temperatures."

Sanford E. Thompson Award—presented by Committee C-9 on Concrete and Concrete Aggregates, for a paper of outstanding merit in the field of concrete and concrete aggregates. This year the award will be made to R. C. Mielenz, L. P. Witte, and O. J. Glantz, of the U. S. Bureau of Reclamation, for their paper entitled "Effect of Calcination on Natural Pozzolans."

The Harold DeWitt Smith Memorial Medal is to be presented at the October meeting of Committee D-13 on Textile Materials to Edward R. Schwarz, Head, Textile Division, Massachusetts Institute of Technology, in recognition of his work in the science of utilization of textile fibers.

Finances

Report for the Fiscal Year of 1950:

The annual statement of the finances of the Society is presented in the form of the report of the auditors for the fiscal year January 1 to December 31, 1950, which appears in Appendix I. The report gives the balance sheet of assets and liabilities, including general funds, building fund, and other special and designated funds; a statement of receipts and disbursements classified into "operating" (budgeted) and "non-operating"; details of the building fund and of special funds; and investments of Society funds.

Receipts and Disbursements.—Total operating receipts were $594,823.78, the largest in the Society's history. Of this amount receipts from dues and entrance fees were $205,580.93 or 34.6 per cent; receipts from sales of publications were $325,997.47 or 54.8 per cent; and receipts from miscellaneous sources consisting principally of advertising, income from investments, income from exhibit and registration fees total $63,245.38 or 10.6 per cent. Receipts from publication sales topped all previous records, which of course is to be expected with the ever-growing activities of the Society and the fact that 1950 included receipts for the first year's sale of the 1949 Book of Standards. Receipts from sales of standards in all forms represented about 83.5 per cent of total publication sales.

Operating disbursements with all current bills paid (as detailed in the auditors' report) and including $40,000 placed in the reserve for the next Book of Standards, were $523,205.74. Of this amount $231,824.77 was spent for publications and $184,249.00 for salaries, comprising 79.4 per cent of the total. (This ratio has averaged over the past decade from about 74 to 82 per cent depending on the volume of annual publications.) The remaining disbursements of $107,131.97 were divided among items of general office expense, expense of technical committees and district councils, meetings, headquarters occupancy (equivalent rent), employees retirement fund, and miscellaneous items.

The difference between operating receipts and disbursements constitutes a favorable operating balance of $71,618.04, which is about 12 per cent of operating receipts. The satisfactory outcome of the year's operations is due principally to the increased volume of publication sales, growth of BULLETIN advertising, and higher income from investments. Increased publication sales follow the normal pattern for the first year following publication of the triennial Book of Standards, in which it is important that there be a substantial favorable operating balance to be applied to the operations of the second and third years of the triennial period, when sales of the Book of Standards fall off. As noted later on, it is anticipated that we shall have to draw upon surplus to balance the 1951 budget.

An informative analysis of current receipts and disbursements can be made on the basis of the "income dollar" and how it was used. Thus, of each dollar of current receipts in 1950,

33.8 cents came from dues,
0.8 cents from entrance fees,
54.8 cents from publication sales,
5.4 cents from advertising,
1.6 cents from investment income,
1.5 cents from exhibit, and
2.1 cents from miscellaneous sources.

We used this income dollar by spending:

34.0 cents for development and publication of standards and reports,
22.6 cents for research, papers, and discussions,
31.4 cents for administration; divided 7.8 cents for membership records and promotion, 10.4 cents for publication sales and promotion, and 13.2 for accounting and other expenses,—leaving
8.3 cents to be applied to reserves, and
3.7 cents over for surplus account available for Society operations.

Balance Sheet.—The auditors' report includes a balance sheet of assets and liabilities. Total assets in all funds were $766,481.06, compared to $602,778.75 a year ago. On the liability side there are listed in the general funds certain accounts payable and certain reserve funds. Concerning the latter, the Board decided that these reserves should be materially augmented, especially the general publication fund and the reserve for the 1952 Book of Standards, where we are faced with increased costs of paper and printing. Accordingly the various reserve funds were increased by a total of $47,000 to the values reported by the auditors. In addition, the Board decided to transfer the sum of $2500 from general surplus to the principal of the ASTM Research Fund, but since this transfer was not actually made before the close of the fiscal year this amount appears as an item of accounts payable.

The report includes a summary of the comparative financial picture of the Society for the past five years.

The surplus in the general funds account on December 31, 1950, was $251,477.84 compared to $219,235.36 a year ago. The ratio of surplus to operating disbursements at current levels is just under 50 per cent.

Building Fund.—The auditors' report includes a statement of cash receipts and disbursements in the Building Fund as well as the balance sheet for the Fund as of December 31, 1950. The cost of the Society headquarters building at the close of the fiscal year was $186,390.87 towards which there has been received a total of $170,042.76 in contributions from members. Other receipts during the life of the Building Fund are shown in the report. The only indebtedness on the building is a 4 per cent note payable to the General Funds of the Society, originally $17,000 in amount but reduced to $12,733.34.

The auditors' report shows that there was paid into the Building Fund from General Funds of the Society during the fiscal year the sum of $5650 as a reserve for depreciation, this constituting a charge in the general budget against headquarters occupancy expense. This added to three previous payments since the inception of the Fund of $14,400 sets up a depreciation reserve of $20,050, so that the Society headquarters are now carried as a depreciated asset of $166,340.87.

Investments.—Investments of Society funds as of December 31, 1950, are given in the auditors' report, both book and market values being shown. As of that date the book value of all investments was $311,843.07, compared with $264,913.38 a year ago; the market value of all investments on December 31, 1950, was $306,220.00, about 2 per cent less than book value.

In view of the inflationary conditions that set in about the middle of 1950 and have since been greatly accentuated, the Board of Directors decided that a greater proportion of the Society's investments should be in sound equities (common stocks) and a lesser proportion in fixed income securities (bonds and preferred stocks). In giving effect to this policy, the Board also decided to purchase stocks of certain open end Mutual Funds as a means of increasing the proportion of common stock holdings. The purchase of Mutual Funds has the important advantage to the Society of obtaining extensive and expert diversification of securities far beyond our ability to accomplish by direct investment with the relatively limited amounts available for this purpose. At the same time, expert management of this portion of the investment portfolio is obtained, but of course at a price represented by the acquisition charge that obtains in the purchase of Mutual Funds.

On the basis of market values as of December 31, 1950, with the holdings in Mutual Funds appropriately divided among the three types of securities, 45.7 per cent of investments were in bonds, 13.3 per cent in preferred stocks, and 41.0 per cent in common stocks.

The following changes during the fiscal year in the investment portfolio are noted:

GENERAL FUNDS

Bought	*Cost*
25 sh. Allis-Chalmers Mfg. Co.	$ 854.55
25 sh. Allis-Chalmers Cum. Conv. Pfd.	2 220.60
75 sh. American Tobacco Co.	5 260.85
10 sh. Carpenter Steel Co. (Stock Dividend)	412.50
25 sh. General Telephone Corp.	637.82
50 sh. Gulf Oil Corp.	3 174.30
40 sh. Lone Star Cement Corp.	3 062.75
15 sh. Montgomery Ward & Co.	832.42
25 sh. Philco Corp.	2 153.15
50 sh. Servel, Inc. Pfd	4 751.70
250 sh. Broad Street Investing Corp.	4 942.50
525 sh. Commonwealth Investment Co.	3 491.25
850 sh. Eaton & Howard Balanced Fund	25 636.00
950 sh. Fundamental Investors, Inc.	17 090.50
	$74 520.89

Sold	*Received*
25 sh. American Can Co.	$ 2 501.30
50 sh. American Cyanamid Co.	2 389.95
10 sh. American Cyanamid Pfd.	1 513.86
100 sh. Atlantic Refining Co.	3 989.03
$8000 Federal Intermediate Credit Bank Debentures (matured)	8 000.00
$3000 Southern Pacific Co. Bonds 1969	2 966.09
$25000 U. S. Treasury Bonds 1954–52	25 210.94
	$46 571.17

RESEARCH FUND

Bought	*Cost*
35 sh. Montgomery Ward & Co.	$ 1 942.30

Matured	*Received*
$2000 Fed. Intermediate Credit Bank Debentures	$ 2 000.00

BUILDING FUND

Bought	*Cost*
$5000 U. S. Savings Bonds, Series G.	$ 5 000.00
1000 sh. Commonwealth Investment Co.	6 650.00
	$11 650.00

COMMITTEE FUNDS

Bought	*Cost*
$5000 U. S. Savings Bonds Series G	$ 5 000.00

No account has been taken, in the accompanying financial statement, of the assets of the Society in the form of publications in stock. An inventory of the principal technical publications as of May 1, 1951, may be summarized as follows:

Year Book (1950)	533
Proceedings (all back copies not incl. 1950)	2 406
1949 Book of ASTM Standards	
Part 1	2 312
Part 2	2 414
Part 3	2 402
Part 4	3 365
Part 5	2 567
Part 6	2 385

1950 Supplements to Book of Standards	
Part 1	3 659
Part 2	3 405
Part 3	[a]
Part 4	3 040
Part 5	4 237
Part 6	2 742
ASTM Methods of Chemical Analysis	5 155
Compilations of Standards	19 788
Selected Standards for Students	30 495
Symposiums and Special Reprints	94 891
Reprints of Standards (Approx.)	155 000
Index to ASTM Standards (1949)	154
Radiographic Standards for Steel Castings	46

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[a] Printing not completed as of May 1, 1951.

1951 Finances:

In preparing the budget for 1951, the Board has estimated current receipts at $544, 000, made up of $208,000 from dues and entrance fees, $285,000 from publication sales, and $51,000 from miscellaneous sources, principally advertising, income from investments, and registration and other fees.

The budget for disbursements has been fixed at $573,000, the two principal items of expense being cost of publications at $237,000 and salaries at $212,000, which together comprise slightly under 80 per cent of the budget. The budget for publication provides for all regular and special publications anticipated in 1951, and for increases in the cost of paper and printing that were imposed at the beginning of 1951. There is also included in the publication budget the sum of $40,000 set aside as reserve for publication of the 1952 Book of Standards, which together with sums set aside in 1950 and a carry-over from previous years provides a reserve of over $100,000 for this purpose.

The salary budget provides for the staff of 54 as of January 1, 1951, together with the three additions thereto mentioned earlier in the report.

It will be seen that the budget as set up anticipates an operating deficit of $29,000 which it is proposed to meet by applying to current operations the sum of $1000 from the Retirement Fund Reserve and $28,000 from Publication Reserve and Surplus, as required.

A review of operations under this budget during the first five months of 1951 indicates that both receipts and disbursements are about as estimated, so that no change in the budget has been considered necessary at this time.

To complete reference to 1951 finances thus far this year, it is noted that during the first five months the following sales and purchases of securities have taken place:

GENERAL FUNDS

Sold	*Received*
$27 000 U. S. Treasury Bonds, 1954–52	$27 194.06
2 000 U. S. Treasury Bonds, 1969–64	2 021.25
1 000 U. S. Treasury Bonds, 1972–67	1 006.57
3 800 U. S. Treasury Bonds, 1972–67	3 941.31
2 000 Hudson Coal Co. Bonds	1 996.87
100 sh. Murray Corp. Pfd.	4 474.54
25 sh. Colgate Palmolive Peet Pfd.	2 373.02
50 sh. Servel, Inc. Pfd.	4 446.10
	$47 453.72

Bought	*Cost*
$20 000 Federal Intermediate Credit Bank Debentures	$20 000.00
45 sh. American Airlines, Pfd.	3 667.55
50 sh. Food Machinery & Chem. Pfd.	4 750.10
50 sh. Chicago, Rock Island and Pacific Pfd.	4 434.60
5 sh. Delaware Power & Light Co.	100.00
33 sh. Philadelphia Electric Co.	833.25
9 sh. Public Service Elec. & Gas Co.	189.00
10 sh. United Gas Improvement Co.	252.50
50 sh. American Cyanamid Co.	3 669.14
50 sh. Aluminum Co. of America	3 679.74
50 sh. Republic Steel Corp.	2 152.93
100 sh. Socony Vacuum Oil Co.	2 510.28
50 sh. Dow Chemical Co.	3 944.32
25 sh. American Tel. & Tel.	3 798.72
100 sh. Baldwin Securities Corp. (dividend)	400.00
1128 sh. Eaton & Howard Balanced Funds	33 997.92
14 sh. Fundamental Investors, Inc. (dividend)	249.76
7 sh. Broad Street Investing Corp. (dividend)	128.52
19 sh. Eaton & Howard Balanced Funds (dividend)	539.41
	$89 297.74

RESEARCH FUND

Sold	*Received*
$2000 Hudson Coal Co. Bonds	$ 1 999.90
30 sh. Butler Bros. Cum. Pfd.	2 446.04
5 sh. Delaware Power & Light Co.	100.00
33 sh. Philadelphia Electric Co.	833.25
9 sh. Public Service Elec. & Gas Co.	189.00
10 sh. United Gas Improvement Co.	252.50
	$ 5 820.69

Bought	*Cost*
280 sh. Broad Street Investing Corp.	$ 5 636.40
100 sh. Jones & Laughlin Steel Corp.	2 636.06
	$ 8 272.46

TEMPLIN FUND

Sold	*Received*
$1000 U. S. Treasury Bonds 1972–67	$1 006.56

Bought	*Cost*
33 sh. Eaton & Howard Balanced Fund	$ 994.62

TOUR FUND

Sold	*Received*
$1500 U S Savings Bonds Series G	$1 422.00

Bought	*Cost*
50 sh. Eaton & Howard Balanced Fund	$1 507.00

BUILDING FUND

Bought	*Cost*
391.297 sh. Commonwealth Investment Co.	$2 816.67

The total book value of Society investments as of May 31, 1951, in all funds was $358,293.79, divided approximately into 31 per cent in bonds, 14 per cent in preferred stocks, and 55 per cent in common stocks. The Society's cash balance in General Funds as of the same date was $283,421.62.

The Board has transferred the sum of $25,000 from Surplus to a new reserve account in General Funds designated Headquarters Building Reserve to provide for increased replacement costs as well as for any needed additions to the headquarters building.

Retirement Plan and Social Security for Employees

During the year there has been a net decrease of one person participating in the ASTM Retirement Plan—three losses through resignations and two additions—bringing the number of participants to 29 out of 40 eligible employees. The initial retirement annuity policies taken out for the two new participants just mentioned are "fully matched" policies, the employees paying premiums of 2½ per cent of salary and the Society matching. For 14 of the 29 participants, including those whose salaries are over $5000, all retirement policies have now been taken out that are required under the Plan.

The Society's annual contribution in premium deposits for retirement policies for the above-mentioned 29 employees is approximately $15,200; the employees' annual contributions, including insurance benefits which most of them have elected, are approximately $3400.

In 1950 the Federal Social Security Law was amended to permit non-profit organizations such as ASTM to participate in Social Security, provided the employer is willing to pay the tax and two-thirds of the employees vote to participate on their part. Believing that it is distinctly to the benefit of the Society's employees for them to be covered by Social Security, the Board of Directors voted to approve participation by the Society, and more than the required two-thirds of the employees voted to participate. Hence as of January 1, 1951, 41 of the then 54 employees entered the Social Security Plan; under the provisions of the law, all new employees thenceforth must participate. In addition, four employees of two subsidiary groups in the Society (the Cement Reference Laboratory and the Joint Committee on X-Ray Diffraction Methods) have also been included. The cost to the Society for 1951 will be approximately $2100.

Thus the Society's annual contribution to the Retirement Plan and Social Security is currently at the rate of $17,300, which is about 3 per cent of estimated current income and 8 per cent of the present salary roll.

The Board of Directors has decided that no change will be made in the provisions of the ASTM Retirement Plan incident to the Society's entry into the Social Security Plan. Therefore the retirement benefits from both plans will accrue to all participants, which materially improves the retirement set-up for the Society employees.

Cooperative Activities

Cooperative relations with other organizations have been maintained during the year in line with established policy, and mention is made here of only a few matters under this category, including certain new developments.

Committees of the Society continue to review Federal materials specifications in draft form, comments and recommendations being forwarded to the Federal Specifications Board for its consideration. Contacts between FSB and ASTM work at the staff level have been maintained.

Four panels set up under the ASTM Ordnance Advisory Committee to pass upon projects submitted by the Ordnance Department of the Army have been at work during the year and have currently reported their opinions and recommendations to the Ordnance Department. These cover plastics, adhesives, rubber, and proposed specifications for steel parts for ordnance materiel.

Committees of the Society are now receiving for informal review and comment preliminary drafts of the Aeronautic Materials Specifications (AMS) as they are developed by the Society of Automotive Engineers.

The Society, acting through the Board, entered into an agreement with the U. S. Bureau of Mines, effective July 1, 1950, under the terms of which the Bureau has undertaken, for a consideration, to do the statistical and clerical work involved in the National Exchange Fuel Groups[1] under the general direction of the Society through the Division of Combustion Characteristics of Committee D-2. The cost of this work is met by the dues of the members of the Exchange Groups and the fees for nonmember participation in the semi-annual fuel exchange projects.

Arrangements have been concluded with the National Bureau of Standards on a modification of the agreement entered into on January 1, 1948, regarding the certification of reference fuels. The Bureau of Standards has included certification of such fuels within the framework of its general certification procedures, at a stipulated fee, leaving to ASTM the certification of such fuels as official "ASTM Knock Test Reference Fuels." Revised procedures for such ASTM certification have been adopted and suitably publicized.

In order to facilitate the coordination of standards being developed by this Society and the American Association of Textile Chemists and Colorists, the Board of Directors has, upon the recommendation of Committee D-13 on Textile Materials, authorized the establishment of a small administrative joint committee of the two organizations and appointed Messrs. H. J. Ball, W. D. Appel, and P. J. Smith as the Society's representatives thereon.

The Board through suitable resolutions has conveyed greetings of the Society to the National Bureau of Standards and the British Standards Institution on the occasion of their Fiftieth Anniversary and extended the Society's felicitations upon the accomplishments of these distinguished institutions during the half century of their existence.

The Board has designated Past-President A. W. Carpenter to represent the Society at the ceremonial session to be held in September, 1951, during the diamond jubilee meeting of the American Chemical Society.

Following are certain appointments on cooperative activities made during the year:

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[1] See ASTM BULLETIN, September, 1950, p. 15, for further information.

A. J. Eickhoff has been appointed as the Society's representative on the Steel Structures Paint Research Council, sponsored by the American Institute of Steel Construction.

F. N. Alquist has been appointed to represent the Society on the Research Council on Causes and Methods of Prevention of Internal Corrosion of Water Pipes, with the understanding that he will report jointly to Committee D-19 on Industrial Water and the Advisory Committee on Corrosion.

L. D. Betz has been appointed to represent Committee D-19 on Industrial Water on the Standard Methods Committee of the Federation of Sewage and Industrial Wastes Associations.

B. W. Scribner has been appointed to succeed R. C. Griffin as an ASTM member of the ASTM-TAPPI Committee on Paper Testing Methods.

L. F. Herron has been appointed to represent the Cleveland District of ASTM on the Joint Building Code Review Committee of Cleveland.

American Standards Assocation:

The Society's technical relationships with the work of the American Standards Association come directly under the responsibility of the Administrative Committee on Standards and the report of that committee (see Appendix II) discusses a number of matters that have developed during the year, including submittal of ASTM standards to ASA for approval as American Standard.

The Board decided to increase the Society's financial support of ASA by naming a third ASTM member to the Standards Council, thus increasing the Society's dues in ASA from $1000 to $1500. This action became effective January 1, 1951, and the Board has appointed J. H. Foote as the new representative on the Standards Council.

The Board is following the progress of the plans of the American Standards Association for "certification under ASA procedures," as such certification might affect ASTM standards that have been approved as American Standard. Certain viewpoints of the Board have been presented to the management of ASA for its consideration.

The Board has expressed its opinion to ASA that as a matter of general policy it believes it is inadvisable for Sectional Committee L22 on Rayon Finished Fabrics to adopt test methods as American Standard that have not been approved by Sectional Committee L14 on Textile Test Methods, of which the Society is a joint sponsor. The Board, however, sees no objection to the inclusion as information in specifications written by L22 of methods of testing that have not yet cleared through L14 and been approved as American Standard, it being the understanding that such methods if published as information do not thereby become formal American Standards. The ASTM representative on L22 has voted in accordance with this ruling and has refrained from voting on the specification requirements of the Specifications for Rayon Finished Fabrics.

The following appointments have been made on certain of the Correlating Committees of ASA: A. G. Ashcroft (reappointed) on Consumer Goods Committee; J. R. Townsend (reappointed) on Mechanical Standards Committee; R. C. Thumser on Chemical Industry Correlating Committee, replacing H. K. Nason.

International Activities:

The principal contacts of the Society with international activities during the past year have been in connection with various standardization projects being undertaken in the International Organization for Standardization (ISO). Brief reference to the status of a number of these projects is contained in the report of the Administrative Committee on Standards (Appendix II), and mention is made here only of the fact that four secretariats for ISO projects that have been assumed by the United States, acting through ASA as the American member of ISO, involve participation in one form or another by ASTM: The secretariat for several subcommittees of ISO Committee 38 on Textiles; the secretariat of ISO Committee 28 on Petroleum Products and Lubricants, functioning through Sectional Committee Z11 of which ASTM is sponsor; the secretariat of ISO Committee 61 on Plastics, where the Society's Committee D-20 on Plastics has been asked to handle the work of this secretariat, which is being done through a special committee consisting of representatives of ASTM, Society of the Plastics Industry, Manufacturing Chemists Association, and the Society of Plastic Engineers, with the understanding that all technical matters will be cleared through Committee D-20; and finally, the secretariat of ISO Committee 26 on Copper and Copper Alloys, which is being handled through a special committee consisting of representatives of various organizations concerned, of which the Society is one.

The Society will be represented at a meeting of ISO Committee 38 on Textiles in England, June 4 to 9, 1951, by Messrs. W. D. Appel and A. G. Scroggie.

The Society continues to maintain contact with the standardizing bodies of certain other countries through exchange of standards for review and exchange of publications. An important piece of cooperation with the British Institution of Petroleum in the development of oil measurement tables is nearing completion.

During the year closing with this annual meeting there were 34 visitors to Society headquarters from twelve foreign countries. While some of these visitors were interested principally in learning about the Society's work in general and how our technical committee activities are carried on, others constituted "technical teams", brought to this country by the Economic Cooperation Administration, which were interested primarily in specific ASTM standardization work. Included in the latter category were groups interested in plastics, rubber, concrete products, and non-destructive testing.

The Society has received from the Economic Cooperation Administration a Certificate of Cooperation in recognition of the assistance given to ECA in this work.

Respectfully submitted on behalf of the Board of Directors,

L. J. MARKWARDT,
President.

C. L. WARWICK,
Executive Secretary.

June, 1951.

APPENDIX I

REPORT OF THE AUDITORS FOR THE FISCAL YEAR JANUARY 1 TO DECEMBER 31, 1950

Philadelphia, January 4, 1951

Mr. C. L. Warwick, *Executive Secretary*
AMERICAN SOCIETY FOR TESTING MATERIALS
Philadelphia, Pennsylvania

Dear Sir:

We have examined the books and accounts of the American Society for Testing Materials for the year ended December 31, 1950. We did not make a detailed audit of all transactions, but made tests to the extent we considered appropriate in determining the accuracy of the accounts. Accounts receivable were not verified by correspondence with debtors. The investments owned by the Society were examined by us; all income from investments for the period under review was properly accounted for.

We have prepared and submit herewith balance sheet as of December 31, 1950, statement of cash receipts and disbursements for the year ended that date, and other supporting schedules which, in our opinion, subject to the foregoing comment, present fairly the financial position of the Society at December 31, 1950, and the results of its operations for the year ended that date.

Respectfully submitted,

John Heins & Co.

BALANCE SHEET AS OF DECEMBER 31, 1950

(Including Special and Designated Funds)

ASSETS

General Funds:			
Cash	$204 957.36		
Less Check No. 3419 as of December 14, 1950 (drawn against cost of 1950 Proceedings and 1950 Supplements to 1949 Book of Standards)	57 000.00		
	147 957.36		
Investments (Market Value $216 791.41)—Cost	222 662.96		
Accounts Receivable	37 458.84		
Note Receivable (Building Fund)	12 733.34		
Advanced to Forest Products Laboratory	500.00		
Total Current Assets General Funds		$421 312.50	
Furniture and Fixtures (depreciated book value)		17 959.00	
Total Assets General Funds			$439 271.50
Building Fund:			
Cash		528.61	
Investments (Market Value $19 741.10)—Cost		20 282.83	
Land (Cost)		30 000.00	
Building and construction costs	156 390.87		
Less Reserve for depreciation	20 050.00	136 340.87	
Total Assets Building Fund			187 152.31
Other Special and Designated Funds:			
Cash: ASTM Research Fund	7 849.31		
Dudley Medal and Marburg Lecture Fund	1 568.15		
Richard L. Templin Award Fund	57.71		
Sam Tour Award Fund	25.75		
Committee Funds	61 659.05	71 159.97	
Investments:			
ASTM Research Fund (Market Value $47 864.11)—Cost	45 772.28		
Dudley Medal and Marburg Lecture Fund (Market Value $5 305)—Cost	6 625.00		
Richard L. Templin Award Fund (Market Value $1 007.19)—Cost	1 000.00		
Sam Tour Award Fund (Market Value $1 426.50)—Cost	1 500.00		
Committee Funds (Market Value $14 084.69)—Cost	14 000.00	68 897.28	
Total Assets, Special and Designated Funds			140 057.25
Total Assets			$766 481.06

LIABILITIES

General Funds:			
Advance Collections	$8 509.65		
Accounts Payable:			
Committee C-1	49.31		
Institute of Petroleum	227.76		
ASTM Research Fund	2 500.00		
Joint Committee on X-ray Diffraction	48 632.87		
Total Current Liabilities, General Funds		$59 919.59	
Life Membership Fund	3 812.50		
Publication Fund	16 841.80		
Book of Standards Reserve	61 900.18		
Executive Retirement Reserve	16 330.00		
Retirement Fund Reserve	8 989.59		
Reserve for Depreciation of Investments	20 000.00	127 874.07	
		187 793.66	
Surplus		251 477.84	
Total Liabilities and Surplus, General Funds			$439 271.50
Building Fund:			
Contributions from Members		170 042.76	
Contributions from ASTM General Funds		3 431.34	
Accumulated Income, Profit on Investments		944.87	
Notes Payable to ASTM General Fund		12 733.34	
Total Building Fund			187 152.31
Other Special and Designated Funds:			
ASTM Research Fund:			
Principal	45 914.60		
Income	7 706.99	53 621.59	
Dudley Medal and Marburg Lecture-Fund:			
Principal	6 625.00		
Income	1 568.15	8 193.15	
Richard L. Templin Award Fund:			
Principal	1 000.00		
Income	57.71	1 057.71	
Sam Tour Award Fund:			
Principal	1 500.00		
Income	25.75		
		1 525.75	
Committee Funds, Unexpended Balances		75 659.05	
Total Special and Designated Funds			140 057.25
Total Liabilities and Funds			$766 481.06

COMPARISON OF GENERAL FUNDS FOR FISCAL YEARS 1946–1950 INCLUSIVE.

At Close of Fiscal Year	Assets				Liabilities			
	Cash	Investments	Accounts Receivable	Furniture and Fixtures	Accounts Payable	Reserve for Book of Standards	Miscellaneous Funds	Surplus
1946	$5 100.03	$160 383.60	$26 150.27	$14 964.50	$7 270.11	$15 000.00	$40 117.54	$144 210.75
1947	3 930.42	163 690.12	37 218.85	16 405.39	7 755.95	20 000.00	36 070.74	157 418.09
1948	50 691.65	191 497.99	46 748.11	17 261.45	7 777.78	40 000.00	31 260.92	227 160.50
1949	56 379.22	192 325.57	36 382.87	18 078.39	12 349.77	40 000.00	31 580.92	219 235.36
1950	147 957.36	222 662.96	50 692.18	17 959.00	59 919.59	61 900.18	65 973.89	251 477.84

RECEIPTS AND DISBURSEMENTS

FOR THE PERIOD JANUARY 1, 1950 TO DECEMBER 31, 1950

Cash balance, January 1, 1950			$56 379.22

RECEIPTS

Operating Receipts (Budgeted):			
Dues and Entrance Fees:			
Current Dues	$195 085.61		
Past Dues	1 786.55		
Advance Dues	3 634.50		
Income, Life Membership Fund	175.00		
Entrance Fees	4 899.27		
Total Dues		$205 580.93	
Sales of publications:			
Book of Standards (Members, additional parts)	38 378.00		
1949 Book of Standards	131 782.04		
1946 Book of Standards and its Supplements	4 348.40		
Methods of Chemical Analysis of Metals	1 750.28		
Compilation of Standards	71 003.96		
Separate Standards	16 713.15		
Selected Standards for Students	7 769.18		
Proceedings	5 620.95		
Spring Meeting Papers	1 337.50		
Bulletin Subscriptions (non-members)	2 948.42		
Special and Miscellaneous Publications	44 345.59		
Total Sales of Publications		325 997.47	
Miscellaneous:			
Advertising—BULLETIN	29 963.35		
Advertising—Index	2 039.68		
Interest and Dividends	9 895.60		
Registration and Other Fees—Meetings	7 327.00		
Annual Meeting Exhibit	8 912.55		
Staff Services	3 896.08		
Miscellaneous	461.12		
Total Miscellaneous Items		62 495.38	
Total Operating Receipts (Budgeted)		594,073.78	
Nonoperating Receipts (Not Budgeted):			
Investments Matured or Sold	46 571.17		
Excess Remittances	2 028.85		
Committee C-1 for Technical Assistant	2 300.00		
Retirement Fund Reserve	2 812.97		
Executive Retirement Reserve	330.00		
Joint AIME-ASTM Publication Fund	122.00		
Institute of Petroleum Publications	323.40		
Sales of X-ray Diffraction Cards	81 615.68		
From Building Fund on Account of Loan	1 133.33		
Annual Meeting Luncheon	946.00		
Miscellaneous	190.00		
Total Nonoperating Receipts		138 373.40	
Total Receipts			732 447.18
Total Receipts for 1950 and Cash Balance, January 1, 1950			$788 826.40

DISBURSEMENTS

Operating Disbursements (Budgeted):			
Publications:			
Year Book	$10 214.50		
1950 Supplements to Book of Standards	32 036.02[a]		
Compilations of Standards	25 945.97		
Separate Standards	5 845.41		
Methods of Chemical Analysis	144.33		
Selected Standards for Students	8 000.00		
Proceedings	28 730.93[a]		
Preprints	6 611.94		
ASTM BULLETIN	31 516.16		
Circulars to Members	2 291.31		
Index to Standards	7 298.59		
Special and Miscellaneous Publications	33 189.61		
Total Disbursements—Publications		$191 824.77	
Salaries	184 249.00		
General Office Expenses	42 191.16		
Expenses—Technical and District Committees	9 950.69		
Expenses—Meetings	10 238.72		
Exhibit—Annual Meeting	2 121.18		
American Standards Association	1 000.00		
Traveling Expenses, Administrative and Special Committees	5 269.22		
Headquarters Occupancy Expenses (includes $5 650 depreciation on building)	17 535.47		
Furniture and Fixtures	2 456.18		
Dues, Contributions, Miscellaneous	517.80		
Legal Services	1 000.00		
Employees' Retirement Fund	14 851.55	291 380.97	
Total Operating Disbursements (Budgeted)		483 205.74	
Nonoperating Disbursements (Not Budgeted):			
Refund of Excess Remittances	2 028.85		
Investments	74 108.39		
Technical Assistant Committee C-1	2 400.00		
Transfer to Joint Committee on X-ray Diffraction	10 323.88		
Cost and Refunds on X-ray Diffraction Cards	33 107.84		
Institute of Petroleum Publications	381.44		
Annual Meeting Luncheon	1 154.75		
Book of Standards from Reserve	33 099.82		
Miscellaneous	1 058.33		
Total Nonoperating Disbursements		157 663.30	
Total Disbursements			$640 869.04
Cash Balance, December 31, 1950			$147 957.36

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[a] These accounts include $57,000 representing a check drawn against cost of the 1950 *Proceedings* and 1950 Supplements to 1949 Book of Standards, but not actually paid on December 31, 1950.

ASTM BUILDING FUND

DETAILS OF BUILDING FUND CASH RECEIPTS AND DISBURSEMENTS FOR THE PERIOD JANUARY 1, 1950 TO DECEMBER 31, 1950

Cash Balance, January 1, 1950			$6 739.02
Receipts			
Contributions from Members		$560.59	
Contributions from ASTM General Funds		1 676.33	
Interest on: U S Treasury Bonds 2½s 1972–67	$212.50		
U S Savings Bonds 2½s Series G	62.50	275.00	
From General Funds for Depreciation on Building, Elevator and Air Conditioning Installation		5 650.00	8 161.92
			14 900.94
Disbursements			
Soundproofing		1 046.00	
Purchase of:			
$5 000 U S Savings Bonds, Series G	5 000.00		
1 000 Shares Commonwealth Investment Co	6 650.00	11 650.00	
Interest on Loan from General Funds		543.00	
Reduction of Loan from General Funds		1 133.33	14 372.33
Cash Balance, December 31, 1950			$528.61

ASTM BUILDING FUND

DETAILS OF ASSETS AND LIABILITIES OF BUILDING FUND AS OF DECEMBER 31, 1950

Assets

Cash in bank				$528.61
Investments: (Market Value $19 741.10)				
$8 500 U S Treasury Bonds 2½s 1972–67			$8 632.83	
5 000 U S Savings Bonds Series G 2½s			5 000.00	
1 000 Shares Commonwealth Investment Co			6 650.00	20 282.83
Property, 1916–18 Race Street, Philadelphia, Pa:				
Purchase Price	$53 625.00			
Commission to Agent	2 681.25			
Settlement Fee, net	197.10			
	56 503.35			
Allocated to:				
Land			30 000.00	
Building		$26 503.35		
Construction Costs:				
Architect's Fee	$11 544.70			
Rent paid to facilitate possession	110.00			
Zoning fees and permits	17.50			
General Contractor	86 933.72			
Air Conditioning	20 749.00			
Elevator	7 548.04			
Air Conditioning Unit	650.75			
Exhaust system for basement	216.25			
Real Estate Agent's Fee	500.00			
Soundproofing	1 953.00			
Miscellaneous	817.48			
	131 040.44			
Less proceeds from sales and salvage of equipment and rentals	1 152.92	129 887.52		
		156 390.87		
Reserve for Depreciation (provided from General Funds)		20 050.00	136 340.87	166 340.87
Total Assets				187 152.31

Liabilities and Funds

Contributions from Members		170 042.76	
Contribution from ASTM General Funds		3 431.34	
Accumulated Income:			
Interest on U. S. Treasury and U. S. Savings Bonds	2 233.28		
Profit on disposal of securities	76.27		
	2 309.55		
Less Interest on Loan from General Funds	1 364.68	944.87	
Note payable to ASTM General Funds		12 733.34	$187 152.31

In addition to the General Funds of the Society, the Treasurer had on hand the following funds as of December 31, 1950:

ASTM Research Fund

	Total	Invested	Uninvested Cash
Principal Account:			
Balance, January 1, 1950	$45 914.60	$45 829.98	$84.62
Receipts:			
Sale of Securities		−2 000.00	+2 000.00
Disbursements:			
Purchase of Securities	———	+1 942.30	−1 942.30
	45 914.60	45 772.28	142.32
Income Account:			
Balance, January 1, 1950		6 376.17	
Receipts:			
Interest and Dividends on Investments	1 580.26		
Sale of Rights	.56	1 580.82	7 956.99
Disbursements:			
Welding Research Council			250.00
Balance, December 31, 1950			7 706.99

Dudley Medal and Marburg Lecture Fund

Balance, January 1, 1950:			
Principal—Investments (at cost)	6 625.00		
Income—Cash	1 509.61	8 134.61	
Receipts			
Interest on Investments		330.00	8 464.61
Disbursements			
Honorarium		200.00	
Dudley Medal		38.50	
Miscellaneous (Travel exp., etc.)		32.96	271.46
			8 193.15
Accounted for as follows:			
Principal—Investments (at cost)		6 625.00	
Income—Cash in bank		1 568.15	8 193.15

Richard L. Templin Award Fund

Balance, January 1, 1950:			
Principal—Investments (at cost)	1 000.00		
Income—Cash in bank	58.96	1 058.96	
Receipts			
Interest on Investments		25.00	1 083.96
Disbursements			
Templin Awards		25.00	
Engrossing Certificates		1.25	26.25
			1 057.71
Accounted for as follows:			
Principal—Investments (at cost)		1 000.00	
Income—Cash in bank		57.71	1 057.71

SAM TOUR AWARD FUND

Balance, January 1, 1950:			
Principal—Investments (at cost)	$1 500.00		
Income—Cash in bank	24.50	$1 524.50	

Receipts

Interest on Investments		37.50	$1 562.00

Disbursements

Tour Award		35.00	
Engrossing Certificates		1.25	36.25
			1 525.75
Accounted for as follows:			
Principal—Investments (at cost)		1 500.00	
Income—Cash in bank		25.75	1 525.75

COMMITTEE FUNDS

Committee A-1 On Steel	956.67	
Committee A-5 On Corrosion of Iron and Steel	1 151.20	
Committee B-3 On Corrosion of Non-Ferrous Metals and Alloys	2 455.58	
Committee B-6 On Die-Cast Metals and Alloys	2 614.81	
Committee B-8 On Electrodeposited Metallic Coatings	11.25	
Committee C-1 On Cement (General Funds)	756.15	
Committee C-1 Cement Reference Laboratory	165.91	
Committee C-9 Sanford E. Thompson Medal Fund	1 034.77	
Committee C-9 On Concrete and Concrete Aggregates	149.82	
Committee C-15 On Manufactured Masonry Units	397.08	
Committee C-18 On Natural Building Stones and Slate	113.76	
Committee D-1 On Paint, Varnish, Lacquer and Related Products	577.80	
Committee D-2 On Petroleum Products and Lubricants	452.89	
Committee D-2 D.C.C. Reference Fuel Account	17 810.12	
Committee D-2 D.C.C. National Exchange Group	1 351.94	
Committee D-2 D.C.C. Equipment Development Project	5 000.00	
Committee D-4 On Road and Paving Materials	6.95	
Committee D-5 On Coal and Coke	1 519.31	
Committee D-7 Pole Research Fund	1 000.00	
Committee D-9 On Electrical Insulating Materials	1 338.03	
Committee D-12 On Soaps and Other Detergents	187.79	
Committee D-13 On Textile Materials	749.90	
Committee D-15 On Engine Antifreezes	43.00	
Committee E-3 On Chemical Analysis of Metals	155.94	
Committee E-9 On Fatigue	198.96	
ASA Sectional Committee on Specifications for Cast Iron Pipe	1 447.01	
Detroit District Council	3 854.05	
Philadelphia District Council	340.13	
Philadelphia District (Annual Meeting Acct.)	439.45	
Pittsburgh District Council	518.35	
Western New York—Ontario District Council	219.67	
Advisory Committee on Corrosion	24 340.81	
Administrative Committee on Ultimate Consumer Goods	202.95	
Joint Committee on X-ray Diffraction	2 809.52	
Joint Committee on X-ray Diffraction Research Associate	1 287.48	
Total Committee Funds		75 659.05
Accounted for as follows:		
Cash in bank and on hand	61 659.05	
Investments (at cost)	14 000.00	75 659.05

INVESTMENTS
AS OF DECEMBER 31, 1950

GENERAL FUNDS

Par Value	Bonds	Cost or Book Value	Market Value[a]
$4 000	Erie RR Co Gen Mtg Income 4½s, 2015	$2 832.00	$3 280.00
2 000	Hudson Coal Co S F 5s Series A 1962	1 697.25	2 000.00
37 600	U S Savings Bonds Series G 2½s	37 600.00	36 417.40
4 800	U S Treasury Bonds 2½s 1972/67	4 800.00	4 834.50
2 000	U S Treasury Bonds 2½s 1969/64	2 000.00	2 024.38
2 000	U S Treasury Bonds 2s 1954/52	2 000.00	2 035.00
25 000	U S Treasury Bonds 2s 1954/52	25 382.81	25 437.50
5 000	Walworth Co 3¼s convertible debentures 1976	5 496.53	4 625.00
		81 808.59	80 653.78
	Preferred Stocks		
Shares			
25	Allis-Chalmers Mfg Co 3¼s cumulative convertible	2 220.60	2 490.63
22	American Airlines, Inc 3½ cumulative convertible	2 244.00	1 776.50
25	Colgate-Palmolive-Peet Co $3.50	2 525.00	2 381.25
100	Murray Corp of America Cumulative 4 per cent	5 050.06	4 612.50
45	Philco Corp 3¾ Series A	4 183.15	4 185.00
50	Servel, Inc	4 751.70	4 487.50
		20 974.51	19 933.38
	Common Stocks		
100	Allis-Chalmers Mfg Co, no par value	4 170.75	4 325.00
100	American Rad & Std Sanitary Corp	1 564.32	1 225.00
100	American Tobacco Co	7 370.97	6 487.50
60	American Viscose Corp	2 323.58	1 815.00
100	Baldwin Locomotive Works	3 822.00	1 362.50
110	Carpenter Steel Co	5 838.50	5 527.50
10	Delaware Power and Light Co.	135.00	210.00
100	General Telephone Corp	3 312.82	2 687.50
100	Gulf Oil Corp	6 273.27	8 025.00
300	International Harvester Co.	9 610.00	9 675.00
100	Lone Star Cement Co.	7 412.17	7 500.00
45	Montgomery Ward Co.	2 646.41	2 970.00
100	Ohio Oil Co.	2 623.35	4 600.00
167	Philadelphia Electric Co	3 519.47	4 342.00
116	Public Service Elec & Gas Co	4 369.50	2 479.50
20	United Gas Improvement Co	315.00	550.00
100	Chase National Bank	3 412.50	3 687.50
		68 719.61	67 469.00
	Mutual Funds		
250	Broad Street Investing Corp	4 942.50	4 672.50
525	Commonwealth Investment Co	3 491.25	3 302.25
850	Eaton & Howard Balanced Fund	25 636.00	24 420.50
950	Fundamental Investors, Inc	17 090.50	16 340.00
		51 160.25	48 735.25
	TOTAL, General Funds	$222 662.96	$216 791.41

ASTM BUILDING FUND

Par Value	*Bonds*	Cost or Book Value	Market Value[a]
$8 500	U S Treasury 2½s 1972–67	$8 632.83	$8 561.10
5 000	U S Savings bonds Series G 2½s	5 000.00	4 890.00
		13 632.83	13 451.10
	Mutual Funds		
Shares			
1 000	Commonwealth Investment Co	6 650.00	6 290.00
	TOTAL, Building Fund	$20 282.83	$19 741.10

ASTM RESEARCH FUND

Par Value	*Bonds*	Cost or Book Value	Market Value[a]
$1 500	B & O RR Gen & Ref 5s Series M, 1996	1 511.25	1 205.63
2 000	Hudson Coal Co S F 5s Series A, 1962	1 697.25	2 000.00
9 900	U S Defense Savings bonds Series G 2½s	9 900.00	9 588.60
2 000	U S Treasury bonds 2½s 1970–65	2 000.00	2 020.00
		15 108.50	14 814.23
	Preferred Stocks		
Shares			
30	Allis-Chalmers Mfg Co 3¼ cumulative convertible	3 007.22	2 988.75
33	American Airlines, Inc. 3½ cumulative convertible	3 366.00	2 664.75
30	Butler Bros., cumulative	3 090.00	2 437.50
12	Consolidated Edison Co of N. Y. $5	1 234.39	1 282.50
29	Consumers Power Co, $4.50	2 914.50	3 219.00
20	Philco Corp., 3¾, Series A	2 030.00	1 860.00
		15 642.11	14 452.50
	Common Stocks		
Shares			
100	Allis-Chalmers Mfg Co	4 421.59	4 325.00
5	Delaware Power and Light Co	67.50	105.00
196	General Motors Corp—gift[b]	6 504.75	9 212.00
55	Montgomery Ward Co	3 151.63	3 630.00
33	Philadelphia Electric Co	583.70	858.00
9	Public Service Elec. & Gas Co	135.00	192.38
10	United Gas Improvement Co	157.50	275.00
		15 021.67	18 597.38
	TOTAL, Research Fund	$45 772.28	$47 864.11

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[a] Market values were taken from current financial publications as of close of market December 27, 1950.

[b] The income from these shares is, by agreement, paid to an annuitant.

DUDLEY MEDAL AND MARBURG LECTURE FUND

Par Value	*Bonds*		
$500	B & O RR Ref & Gen Mtg 6s, Series J, 1995	$550.00	$445.00
6 000	B & O RR Ref & Gen Mtg 5s, Series G, 1995	6 075.00	4 860.00
		6 625.00	5 305.00

RICHARD L. TEMPLIN AWARD FUND

Par Value		*Cost or Book Value*	*Market Value*[a]
$1 000	U S Treasury Bonds 2½s, 1972–67	1 000.00	1 007.19

SAM TOUR AWARD FUND

$1 500	U S Savings Bonds, Series G 2½s	1 500.00	1 426.50

COMMITTEE FUNDS

$1 000	U S Treasury Bonds 2⅞s 1960–55	1 000.00	1 071.25
2 000	U S Treasury Bonds 2½s 1969–64	2 000.00	2 021.88
3 000	U S Treasury Bonds 2½s 1970–65	3 000.00	3 030.00
3 000	U S Treasury Bonds 2½s 1972–67	3 000.00	3 021.56
5 000	U S Savings Bonds Series G 2½s	5 000.00	4 940.00
		14 000.00	14 084.69

GRAND TOTALS, Bonds	133 674.92	130 742.49
GRAND TOTALS, Preferred Stocks	36 616.62	34 385.88
GRAND TOTALS, Common Stocks	83 741.28	86 066.38
GRAND TOTALS, Mutual Funds	57 810.25	55 025.25
	$311 843.07	$306 220.00

APPENDIX II

REPORT OF ADMINISTRATIVE COMMITTEE ON STANDARDS

In pursuance of its principal function, the Administrative Committee on Standards has acted upon numerous tentatives and revisions which have been submitted since the Society's Annual Meeting in June, 1950. Hereinafter is included a complete record of such recommendations as have been approved.[1] These actions included 44 new tentatives accepted during the year as well as revisions of 58 tentatives, tentative revisions of 22 standards, 12 standards reverted to tentative, and 2 tentatives withdrawn. A number of these were acted upon at a meeting of the committee held on September 26, 1950, and the remainder were handled by correspondence.

The committee has continued its efforts toward improvement of quality and form of standards. At the instance of the committee, the Board of Directors has arranged with Committee E-1 on Methods of Testing for preparation of a manual on the form of ASTM methods for guidance of the technical committees. It is planned that when this manual is completed it will be put in the hands of subcommittee and group leaders engaged in the writing of methods of testing as well as in the hands of technical committee chairmen and officers. It is hoped that this procedure will insure more adequate treatment of certain important features such as editorial form, accuracy, reproducibility, etc.

In the report a year ago, attention was called to an addition that was being made to the regulations governing the operation of the Administrative Committee on Standards of a procedure that would provide for advising those having a negative viewpoint of their privilege of being present at meetings of the Administrative Committee at which recommendations of concern to them were being considered. While this was already the general practice followed, it was felt desirable to formalize the practice by having the procedure set forth in the regulations. In making this addition, a complete review of the regulations was carried through and the regulations brought up to date.

Several technical committees of the Society have found need for some special provisions to be made in their standards in view of the present emergency conditions. It is accordingly being proposed that an emergency procedure somewhat along the lines of that used during the recent war be instituted. In anticipation of consideration of this on the part of the Board, the Administrative Committee has reviewed the procedure that had been previously in effect, as a result of which certain recommendations, looking toward having a more adequate review of these emergency procedures, have developed.

During the year the committee has also studied several projects looking to the establishment of new technical committees in the Society. Some of these investigations have resulted in setting up new committees and others are still in process of organization.

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[1] This list is not reproduced here, since the recommendations have all been announced currently throughout the year in the ASTM BULLETIN.—Ed.

EXPANSION OF STANDARDIZATION ACTIVITIES

Of the projects referred to in the report of a year ago, organization has now been effected of the following committee:

D-22 on Methods of Atmospheric Sampling and Analysis.

It is expected that the organization meeting of the Committee on Radioactive Isotopes (E-10) will be held during the present Annual Meeting.

Cellulose and Cellulose Derivatives.—A Committee on Cellulose and Cellulose Derivatives has been authorized by the Board and the personnel is now being assembled. The recommendation to organize this new committee resulted from a conference of the various interested groups which was held in November, 1950. The committee is being given the designation D-23.

Smooth Surface Organic Floor Coverings.—The study mentioned in the report of a year ago with respect to the Society organizing a new committee to deal with asphalt floor tile and other smooth surface floor coverings is still under way.

Industrial Diamonds.—The proposal had been made that the Society organize a committee to deal with industrial diamonds. In exploring this subject, there developed the importance of having work done in the field possibly including all cutting and abrasive materials, but also that the subject should be developed from a somewhat broader point of view, and rather than to consider industrial diamonds *per se*, the tools involved, should also be included. It appeared to be too broad a subject for the ASTM to sponsor, and the subject was accordingly referred to the American Standards Association with the thought that it might wish to explore it further.

Other Pending Proposals.—At various times inquiries have been received and proposals have been submitted with respect to the Society undertaking work in the field of fertilizers, ore sampling, surface chemistry, mass spectroscopy, and, more recently, standards dealing with the newer jet alloys. All of these subjects will require further development.

RELATIONS WITH AMERICAN STANDARDS ASSOCIATION

Two methods under the procedure of the American Standards Association are used by ASTM in submitting recommendations to the ASA. One is a combination of the so-called existing standards procedure and the proprietary sponsorship procedure. On the initial reference of an existing ASTM standard to the ASA, it is submitted under the existing standards procedure with a supporting statement giving a history of the development of the standard and indicating the degree of its acceptance. If the standard is approved as American Standard, the ASTM is granted proprietary sponsorship so far as any future revisions are concerned, and such revisions are brought to the attention of the ASA currently for approval under this proprietary sponsorship procedure.

The second method consists of having standards reviewed in a sectional committee made up of representatives of a number of interested organizations but with ASTM as sponsor or co-sponsor for the sectional committee.

Standards Submitted Under the Existing Standards Procedure:

No new standards were submitted during the past year under the existing standards procedure.

Revised Standards Submitted Under Proprietary Procedure:

In view of the Society having been designated as proprietary sponsor for the revisions of ASTM standards that had been approved as American Standard under the existing standards procedure, it has submitted to the American Standards Association during the year revisions of 1 specification relating to zinc-coated iron or steel sheets, 3 relating to copper and brasses, 8 relating to gypsum, 4 relating to structural building shapes, and 1 relating to pigments.

Standards Submitted Under Sectional Committee Procedure:

During the year reports were received from six of the sectional committees for which the Society is sponsor or joint sponsor as follows:

Sectional Committee A37 on Road and Paving Materials.—This committee has recommended the approval as American Standard of 4 ASTM methods and 1 ASTM specification, and the approval of revisions of 19 others that already had been approved as American Standards.

Sectional Committee A88 on Magnesium Oxychloride Cement Flooring.—This committee has recommended the approval as American Standard of 4 specifications for oxychloride cement flooring.

Sectional Committee C59 on Electrical Insulating Materials.—This committee has recommended the approval as American Standard of 5 ASTM methods and 1 ASTM specification, and the approval of revisions of 2 American Standards.

Sectional Committee Z11 on Petroleum Products and Lubricants.—This committee has recommended the approval as American Standard of 4 ASTM methods, and the approval of revisions of 3 American Standards.

Sectional Committee Z23 on Specifications for Sieves for Testing Purposes.—This committee has recommended the reaffirmation as American Standard of the Specifications for Sieves for Testing Purposes (Wire Cloth Sieves, Round-Hole and Square-Hole Screens or Sieves) (ASTM E 11 - 39; ASA Z23.1-1939).

All of these reports have been referred to the American Standards Association. Action is still pending so far as the recommendations of Sectional Committee A37 are concerned; the others have been approved.

INTERNATIONAL RELATIONS

The Society continues to receive from other countries standards with the request for comment. ASTM committees review these standards and frequently suggestions result. Apart from such informal cooperation, however, quite a number of proposals have been received through the American Standards Association concerning projects that have been proposed under the International Organization for Standardization (ISO):

Textiles.—The general secretariat for ISO Committee 38 has been assigned to Great Britain, but the United States holds the secretariat for several subcommittees. During the past year American participation in ISO Committee 38 has been handled in a special subcommittee of Sectional Committee L14 on Textile Test Methods. It was felt, however, that American industry's interest in this work was considerably broader than might be expected to be represented under a committee organized primarily to cover test methods. It would also seem preferable to have American industry represented directly in a matter of this sort rather than indirectly as would be the case under Sectional Committee L14. Accordingly an American group is being organized under the joint sponsorship of ASTM and the American Association of Textile Chemists and Colorists to consist of representatives of all American interests concerned.

A meeting of ISO Committee 38 is being held in Bournemouth, England, June 4–9, 1951, and a rather extensive delegation from the United States is going abroad to attend the meeting, W. D. Appel and A. G. Scroggie being the official ASTM delegates.

Petroleum Products.—The American Standards Association as the official American member of ISO has asked Sectional Committee Z11, of which ASTM is sponsor, to handle the secretariat for ISO Committee 28. The American petroleum industry has raised funds to support the work of the secretariat for one year. A committee of API will function in the handling of the duties of this secretariat.

Rubber.—The secretariat for ISO Committee 45 is held by Great Britain. The United States was host to the ISO Committee for its last meeting which was held in Akron, Ohio, in October, 1950. A number of test procedures relating to rubber, where international discussions seem to have value, came up for consideration, as discussed in the annual report of Committee D-11 on Rubber Products.

Viscosity.—The work of the ISO Committee on Viscosity is going forward and America is being asked to assume the secretariat. The manner of organization to handle the secretariat is now under discussion.

Plastics.—America has accepted the secretariat for ISO Committee 61 on Plastics. The American Standards Association, which is the official American member in ISO, has requested ASTM Committee D-20 on Plastics to handle the work of this secretariat, and Committee D-20 has organized a special committee consisting of representatives of ASTM, Society of the Plastics Industry, Manufacturing Chemists Assn., and the Society of Plastics Engineers. Any technical matters will be cleared through Committee D-20. It is proposed to hold a 1- or 2-day meeting of the ISO Committee in America next fall in conjunction with the meeting of the International Union of Pure and Applied Chemistry, at which time it is planned to give consideration to several test methods as well as to the subject of nomenclature.

Shellac.—Subcommittee XIII on Shellac of ASTM Committee D-1 has been set up as the American committee in charge of contacts with the ISO Committee on Shellac (No. 50) for which India holds the secretariat, and has submitted a number of comments to the ISO Committee with respect to standards dealing with shellac that are now under consideration. A meeting of the ISO Committee was held in Delhi, India, January, 1950.

Solid Mineral Fuels.—ASTM Committee D-5 on Coal and Coke is being looked to to handle American contacts with this ISO Committee No. 27. A meeting of the ISO Committee is proposed for December, 1951.

Iron and Steel.—Committee A-1 on Steel has indicated that it favors American participation in the work of this ISO Committee No. 17 as an observer.

Chemistry.—The following ASTM committees have indicated an interest in participating in the ISO Project on Chemistry:

- D-1 on Paint, Varnish, Lacquer, and Related Products
- D-3 on Gaseous Fuels
- D-12 on Soaps and Other Detergents.

Laboratory Glassware.—Committee E-1 on Methods of Testing has indicated that it favors American participation in the work of this ISO Committee No. 48 on the basis of an observer.

General Definitions Relating to Chemical and Physical Test Results.—Committee E-11 on Quality Control has indicated that it favors American participation in the work of this ISO Committee (No. 69).

Mica.—Subcommittee IX on Mica of ASTM Committee D-9 has been set up as the American committee in charge of contacts with the ISO Committee on Mica (No. 56) for which India holds the secretariat, and has submitted a number of comments to the ISO Committee with respect to standards dealing with mica that are now under consideration. A meeting of the ISO Committee was held in Delhi, India, in January, 1950, at which America was represented by Messrs. U. C. Lal and Peter DeCicco, representatives in India of the Asheville-Schoonmaker Mica Co.

Copper and Copper Alloys.—America holds the secretariat for the ISO Committee on Copper and Copper Alloys (No. 26), and American participation is being arranged through a special group consisting of representatives from various organizations concerned. Both Committees B-2 and B-5 of ASTM are interested in this subject.

Paper.—ASTM Committee D-6 on Paper and Paper Products has indicated that it favors American participation in the work of this ISO Committee on the basis of an observer.

Timber.—ASTM Committee D-7 has indicated that it favors American participation in the work of the ISO Committee on Timber (No. 55).

Respectfully submitted on behalf of the committee,

L. B. JONES,
Chairman.

R. E. HESS,
Secretary.

APPENDIX III

REPORT OF ADMINISTRATIVE COMMITTEE ON RESEARCH

The Administrative Committee on Research held one meeting during the year at Society Headquarters on December 4, 1950.

The usual contribution of $250 to the Welding Research Council in support of researches of value to ASTM was made. The committee also approved a request of Committee E-5 on Fire Tests of Materials and Construction for a grant of approximately $450 from the ASTM Research Fund to obtain a compilation and evaluation of flame spread data at the National Bureau of Standards. As a result of this grant a valuable report on this subject was prepared for Committee E-5 by S. H. Ingberg, formerly with the Bureau.

The committee has noted with interest the excellent progress that is being made by the Advisory Committee on Corrosion towards the raising of $100,000 to support a 10-yr program for ASTM exposure test sites, over 80 per cent of this sum having now been raised. The use of these test sites by committees of the Society in the non-metals fields is being encouraged.

The committee has also followed with interest the discussions in two other committees of the Society concerning the establishment of research programs, namely, Committee C-16 on Thermal Insulating Materials for a project to study the effect of moisture on thermal conductivity of certain materials, and Committee D-7 on Wood for a project to study full-size testing of wood poles. At the suggestion of the Research Committee, Committee C-16 has brought its research program to the attention of certain other ASTM committees that might be interested.

An extensive research program is now getting under way in the Joint Committee on Effect of Temperature on the Properties of Metals sponsored by ASME and ASTM for which the committee is going to raise approximately $85,000. These researches will cover the general accumulation of data on high-temperature properties of metals, including some of the newer compositions of special interest to the aircraft industry and in the building of gas turbines. Proposals made to the Research Committee by our Committee A-3 on Cast Iron for studies of the high-temperature properties of that material were taken up with the Joint Committee with the result that a program for research in this field is being included in the over-all program of the Joint Committee.

The research associateship at the National Bureau of Standards supported by the Joint Committee on Chemical Analysis by X-Ray Diffraction Methods has been continued into its third year. Through this associateship the Joint Committee is carrying out investigations to improve the accuracy of existing X-ray diffraction data and extending the work into certain new fields. This work is supported by funds of the Joint Committee received through sales of X-ray Diffraction Data Cards and by contributions from industry. As part of this whole project, work on the compiling and tabulation of data for new X-ray Cards is being carried out at Pennsylvania State College on funds of the Joint Committee.

During the year the Research Committee has continued its review of the manner in which the technical committees of the Society are organized for committee work in the field of research. A revised summary of the forms of organization was reviewed by the committee at its meeting last December, and copies have been circulated to the officers of technical committees for their information and correction or addition where need be.

Some progress has been made during the year on the plan of compiling and publishing statements of unsolved problems in the testing of materials prepared by committees of the Society. As reported a year ago, the Research Committee drew up a pattern or form for the preparation of formal statements of such problems. Some 35 such statements have been submitted by eight committees and are now being edited for publication. In addition to publication of suitable reference thereto in the ASTM BULLETIN, it is planned to send copies of these statements to the deans of engineering and science schools inviting their consideration of these projects as being desirable topics for undergraduate or graduate research and offering to supply additional copies for the use of various engineering departments in universities and colleges. It is planned also to send copies to the heads of various research institutes. The committee plans to defray the expenses for this work from the income of the Research Fund.

The committee plans to publish in the ASTM BULLETIN early in 1952 a complete report of all ASTM research projects, bringing up to date the previous such report published in the May, 1947, BULLETIN.

The Research Committee has continued for the Society its contact with the National Research Council, Division of Engineering and Industrial Research. Two activities of particular interest to the Society are the Highway Research Board and the Building Research Advisory Board of that Division.

Respectfully submitted on behalf of the committee,

W. C. VOSS,
Chairman.

C. L. WARWICK,
Secretary.

APPENDIX IV

REPORT OF ADMINISTRATIVE COMMITTEE ON PAPERS AND PUBLICATIONS

The activities of the Administrative Committee on Papers and Publications have consisted largely of reviewing material for publication and planning for its publications, as well as planning the technical program for the Annual Meeting. Accordingly, this report is largely a record of the various regular and special publications that have been issued during the past year.

As a result of the First Pacific Area National Meeting held in San Francisco in October, 1949, there were published during the past year 6 separate technical publications totalling around 511 pages. In addition, 15 papers presented at this meeting were published in various issues of the ASTM BULLETIN totalling approximately 112 pages, and 6 papers will appear in the 1950 Proceedings comprising 130 pages. (Several papers presented at the San Francisco meeting are still to appear in the Symposium on Triaxial Shear Testing now in preparation.)

These publications are in addition to the papers and symposiums presented at the 1950 Annual Meeting. The number of technical papers being handled by the Society is increasing every year, as is evidenced by the large number of papers published in the Proceedings, the increasing number of special technical publications issued each year, and the fact that although the number of issues of the BULLETIN has been increased from 6 to 8 a year, the number of pages devoted to technical papers in each issue remains the same (approximately 40 pages).

It may be of interest to note that apart from the regular publications such as Proceedings and Book of Standards, 35 publications were issued by the Society during the past year (including 8 compilations issued during the first half of this year). Also, 2 reprints of publications have been struck off as listed later in this report.

The Society has been faced with an increase in printing cost this year, and in view of the amount of material to be printed every effort is being made toward condensation without loss of essential details.

The Manual for Authors of ASTM Papers which was mentioned in last year's report as being in course of preparation is now available. This has proved of considerable value. It has been welcomed by the authors and has resulted in the preparation of better manuscripts. There has been quite a bit of interest expressed in this Manual on the part of universities and in some of the larger industrial organizations where individuals are engaged in writing technical reports.

RECORD OF PUBLICATIONS ISSUED DURING THE YEAR

Regular Publications:

1950 Proceedings, 1480 pp, 7100 copies. Available May, 1951.

1950 Supplements to the Book of Standards
- Part 1, 326 pp, 10,600 copies.
- Part 2, 236 pp, 9350 copies.
- Part 3, 370 pp, 8500 copies. Available May, 1951.
- Part 4, 352 pp, 8000 copies.

Part 5, 598 pp, 8000 copies.

Part 6, 297 pp, 7650 copies.

A.S.T.M. Methods for Chemical Analysis of Metals, 486 pp, 7200 copies.

1950 Year Book, 588 pp, 6900 copies.

1950 Index to ASTM Standards, 274 pp, 22,000 copies. Available May, 1951.

ASTM BULLETIN, 8 issues (May, 1950, to April, 1951) total number of pages 772, average number of copies 10,890.

A statement of the volume of the principal publications for the past four years is given below:

Publications	1947 Pages	1948 Pages	1949 Pages	1950 Pages
Proceedings	1206	1366	1252	1480
Book of A.S.T.M. Standards	....	....	8396	....
Supplements to Book of A.S.T.M. Standards	2015	1648	....	2179
A.S.T.M. Methods of Chemical Analysis of Metals	....	....	....	486
Index to A.S.T.M Standards	254	272	280	274
Year Book	542	569	570	588
Spring Meeting Symposiums	119	....	....	....
ASTM BULLETIN	668	664	728	772
	4804	4519	11226	5779

Special Compilations of Standards Published from April 15, 1950 to April 15, 1951:

A.S.T.M. Specifications for Rolled Structural Steel, 40 pp, 2000 copies.

A.S.T.M. Specifications for Carbon & Alloy-Steel Bars, 60 pp, 1200 copies.

A.S.T.M. Standards on Wires for Electrical Conductors (B-1), 200 pp, 1000 copies.

A.S.T.M. Standards on Copper and Copper Alloys (B-5), 542 pp, 1100 copies.

A.S.T.M. Standards on Petroleum Products and Lubricants (D-2), 784 pp, 7500 copies.

A.S.T.M. Standards on Gaseous Fuels (D-3), 144 pp, 1200 copies—not yet issued, available May, 1951.

A.S.T.M. Standards on Rubber and Rubber-Like Materials (D-11), 670 pp, 1800 copies—not yet issued.

A.S.T.M. Standards on Textile Materials (D-13), 584 pp, 2700 copies.

A.S.T.M. Standards on Benzene, Toluene, Xylene, Solvent Naphtha, 60 pp, 1000 copies.

A.S.T.M. Standards on Industrial Waters (D-19), 168 pp, 1000 copies.

A.S.T.M. Procedures for Testing Soils (D-18), 426 pp, 5000 copies.

A.S.T.M. Standards for Students in Engineering, 320 pp, 35,000 copies.

A.S.T.M. Manual on Measurement and Sampling of Petroleum and Petroleum Products (D-2), 134 pp, 2000 copies.

A.S.T.M. Manual on Quality Control of Materials (E-11, STP 15-C), 141 pp, 5000 copies.

1950 Appendix to ASTM Manual of Engine Test Methods for Rating Fuels, 8 pp, 2000 copies.

Charts:

Single Panel Paint Record Form, 1 p, 10,000 copies.

Multi-Panel Paint Record Form, 1 p, 10,000 copies.

Special Publications Also Issued:

1950 Edgar Marburg Lecture.—"Chemical Spectroscopy" by Wallace R. Brode, 50 pp, 2000 copies.

Symposium on Evaluation Tests for Stainless Steels (STP 93), 237 pp, 3000 copies.

Papers on Radiography (STP 96), 64 pp, 2000 copies.

Symposium on Rapid Methods for Identification of Metals (STP 98), 83 pp, 2000 copies.

Symposium on Use of Pozzolanic Materials in Mortars and Concretes (STP 99), 211 pp, 3000 copies.

Symposium on Ultrasonic Testing (STP 101), 139 pp, 2000 copies.

Symposium on High Additive Content Oils (STP 102), 67 pp, 2000 copies.

Symposium on Application of Statistics (STP 103), 31 pp, 2500 copies.

Symposium on Dynamic Stress Determinations (STP 104), 65 pp, 3000 copies.

Symposium on Turbine Oils (STP 105), 56 pp, 2000 copies.

Symposium on Plasticity and Creep of Metals (STP 107), 72 pp, 2500 copies.

Symposium on Corrosion of Materials at Elevated Temperatures (STP 108), 127 pp, 3000 copies.

Symposium on the Nature, Occurrence, and Effect of Sigma Phase (STP 110), 187 pp, 2000 copies.

Symposium on the Role of Non-Destructive Testing in the Economics of Production (STP 112), 163 pp, 2500 copies.

Symposium on Identification and Classification of Soils (STP 113), 96 pp, 2500 copies.

Report on the Strength of Wrought Steels at Elevated Temperatures, by R. F. Miller and J. J. Heger (STP 100), 113 pp, 1600 copies.

Physical Constants of Hydrocarbons Boiling Below 350 F (STP 109), 18 pp, 2500 copies.
Data on Corrosion and Heat-Resistant Steels and Alloys—Wrought and Cast (STP 52-A), 84 pp, 2000 copies.
1949 Supplement to the Bibliography and Abstracts on Electrical Contacts (STP 56-F), 29 pp, 1200 copies.
Report on Standard Samples for Spectrochemical Analysis (STP 58-B), 35 pp, 2000 copies.
1950 Supplement to the Metal Cleaning Bibliographical Abstracts (STP 90-A), 31 pp, 2000 copies.
Alphabetical and Numerical Indexes of X-Ray Diffraction Patterns (STP 48-B), 747 pp, 700 copies.
X-Ray Diffraction Data for Chemical Analysis, Second Supplement (STP 48-B), 1337 cards, 1000 sets.
Revised Original set and 1st Supplement, 2723 cards, 1000 sets.

Reprints of Publications:

Symposium on Colorimetry and Photometry, 74 pp, 500 copies.
Radiographic Standards for Steel Castings, 35 negatives, 45 sets.

Special Publications in Prospect:

ASTM-IP Manual of Oil Measurement Tables (STP number not yet assigned), 544 pp, 5000 copies.
Monograph on Paper and Paper Products (D-6)
Monograph on the Significance of Tests of Textile Materials (D-13)
Manual on Engine Antifreezes (D-15)
Manual on Industrial Water (D-19)
Procedures for Emission Spectrochemical Analysis (E-2)
Creep Data on Austenitic Steels (Joint Com. on Effect of Temperature)
Bibliography on Electrical Contacts (STP 56), 250 pp, 1500 copies. This will be accumulative and will supersede previous editions of supplements.
Symposium on Triaxial Testing of Soils and Bituminous Materials (STP 106), 170 pp, 3000 copies.
Symposium on Measurement of Viscosity (STP 111), 50 pp, 2000 copies.
Symposium on Thermal Insulating Materials (STP 114), 80 pp, 2000 copies.

CURRENT ACTIVITIES

A full program is in prospect for the 1951 Annual Meeting with a number of symposiums scheduled as follows:

Symposium on Consolidation Testing of Soils—under the auspices of Committee D-18 on Soils for Engineering Purposes.
Symposium on Surface and Subsurface Reconnaissance—under the auspices of Committee D-18 on Soils for Engineering Purposes.
Symposium on Bulk Sampling—under the auspices of Committee E-11 on Quality Control of Materials.
Symposium on Structural Sandwich Construction—under the auspices of Committee C-19 on Structural Sandwich Construction.
Symposium on Acoustical Materials—under the auspices of Committee C-20 on Acoustical Materials.
Symposium on Flame Photometry—under the joint auspices of Committee C-1 on Cement and D-2 on Petroleum Products and Lubricants.
Symposium on Measurement of Consumer Wants—under the auspices of the Administrative Committee on Ultimate Consumer Goods and arranged with the aid of the Social Science Research Council.

In addition to the above, there are a number of other technical papers which will be presented at the Annual Meeting. A number of papers are being offered to the Society for publication but not presentation at a meeting. These for the most part will be published currently in the ASTM BULLETIN or in the Proceedings.

Respectfully submitted on behalf of the committee.

C. L. WARWICK,
Chairman.

APPENDIX V

REPORT OF ADMINISTRATIVE COMMITTEE ON ULTIMATE CONSUMER GOODS

The Administrative Committee on Ultimate Consumer Goods held a meeting on January 25, 1951, at the offices of the United States Testing Co., Hoboken, N. J.

The Administrative Committee is pleased to report that all arrangements have been completed for a Symposium on Consumer Want Determination to be held at the Annual Meeting of the Society in June, 1951. Dr. Paul S. Olmstead, who is in charge of the program, has arranged to have the practical approach presented in two papers by officers from the Quartermaster Corps describing methods and results which have been used by the Army. The theoretical approach will be presented in three separate papers dealing with the merits and limitations to be expected from the use of questionnaires, panels, and depth interviews, respectively.

Mention should be made of, what to the members of the Administrative Committee appears to be a significant trend, namely, the increasing number of instances in which advice and comment has been sought from the members of this committee's Advisory Group on Consumer Want Determination, headed by Dr. Olmstead. The Administrative Committee believes it is desirable from the standpoint of the ASTM to encourage this trend as there is need for a group which may become generally recognized as an authority in this field of research. The Administrative Committee is planning to promote this idea by giving publicity to the work of the Advisory Group through the medium of an article in the ASTM BULLETIN. A. G. Ashcroft has also included reference to its functions in a paper he will present at the annual conference of the British Textile Institute in Brighton, England, on May 25, 1951.

In the belief that one function of the Administrative Committee is to supply technical committees which choose to do work on consumer standards with pertinent information, the committee has approved a project to define and classify the various types of consumer standards as they exist today. Classification would be by such group names as industrial, minimum, mandatory, etc. A subgroup, composed of members of the Society who are thoroughly familiar with the ASTM standards and who have an interest in this subject, is being formed to undertake the project.

The Administrative Committee considers that the interests of the ASTM will best be served by the reappointment of A. G. Ashcroft as the Society's representative on the ASA Consumer Goods Committee, and so recommends.

Respectfully submitted on behalf of the committee,

HERBERT J. BALL,
Chairman.

R. E. HESS,
Secretary.

APPENDIX VI

REPORT OF ADMINISTRATIVE COMMITTEE ON SIMULATED SERVICE TESTING

The last meeting of the Administrative Committee on Simulated Service Testing was held at Society Headquarters on October 3, 1950, with but one absentee. At this meeting were present the several ASTM staff members whose intimate contact with the many technical committees of the Society proved to be of great value to the committee.

Of the several subjects which had previously been discussed from the standpoint of possible Society action, the following recommendations were made:

Gaskets.—That Consideration be given to the desirability of establishing a separate committee on gasket materials.

Soldered Joints.—Inasmuch as the American Welding Society was organizing a committee on this subject, and is soliciting suggestions for their guidance, it would seem advisable to maintain close liaison with this effort and not pursue this subject in ASTM for the time being.

Humidity Testing.—After having determined the lack of interest or ability of several committees to pursue this work, a specific request was made to Committee E-1 on Methods of Testing that it undertake such an assignment.

Stress Corrosion Cracking.—The Bureau of Standards has now initiated a research project in this field, and several ASTM committees and members have rendered assistance in getting this project working. The initial work will be on brass (season cracking).

Among the several items discussed, which are in the exploratory state, are the following:

1. Publication of a paper on Fracture Tests for Steel.
2. Extension of activities in the field of low-temperature properties.
3. Symposium on Bearing Metals and Lubricants.
4. Symposium on Fretting Corrosion or Friction Oxidation.
5. Bend Testing.
6. Pressure Testing (high pressure, 25,000 psi, etc.)

Inasmuch as the master list of suggested considerations for the committee is about five years old, an up-to-date list is now being compiled and will form the basis of discussion at the next meeting of the committee.

Respectfully submitted on behalf of the committee,

L. L. Wyman,
Chairman.

R. E. Hess,
Secretary.

REPORT OF COMMITTEE A-1

ON

STEEL*

While no particularly significant trend in Committee A-1 activities occurred during the past year, there has been a great amount of consideration given to the some 150 specifications and tests under the committee's jurisdiction and quite a few new specifications and tests are in their final stages. A mid-winter series of meetings of the committee were held in Cleveland, Ohio, on February 5 to 7, inclusive. Despite the railroad strike, these meetings were among the best attended in recent years.

The development of mechanical testing procedures applicable to the various product fields has continued, with procedures for structural steel and tubular products now being considered for publication by the main committee.

There have been several new specifications for low-temperature service materials developed. The Tentative Specifications for Seamless and Welded Steel Pipe for Low-Temperature Service (A 333 – 50 T) were approved for publication by the Administrative Committee on Standards on January 25, 1951. Also in their final stages and being balloted upon by Committee A-1 are Proposed Tentative Specifications for Steel Tubes for Low-Temperature Service. Proposed Tentative Specifications for Ferritic Steel Castings for Pressure-Containing Parts Suitable for Low-Temperature Service are expected to be proposed for publication during 1951.

Among the very significant actions taken during the year was the request that the Society issue, as soon as possible, for certain of the alloy steel tubular specifications, emergency provisions permitting a higher phosphorus content. The producers, who must use scrap which may be high in phosphorus, stressed the necessity for emergency action in raising the permissible limits on check analysis to 0.045 per cent, maximum. This action would help eliminate sacrifice of important alloys in those cases where it is extremely difficult to lower the phosphorus content to the present required limit.

Discharge of Subcommittee.—The Special Subcommittee on Rolled Carbon-Steel Locomotive Frames was discharged during the year because of completion of its work. No comments suggesting changes have been received on the Tentative Specifications for Rolled Carbon-Steel Locomotive Frames (A 287 – 46 T).

Personnel.—While there have been many changes in personnel, the final analysis shows practically the same total membership as in 1950. The committee consists of 272 voting members, including 130 producers, 105 consumers, and 37 general interest members.

Following the resignation of W. Warner of the Inland Steel Co. as vice-chairman of Subcommittee XI on Steel for Boilers and Pressure Vessels, R. W. Mooty of the Tennessee Coal, Iron and Railroad Co. was appointed in his place.

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* Presented at the Fifty-fourth Annual Meeting of the Society, June 18–22, 1951.

H. C. Larson of the Bethlehem Steel Co., former secretary of Committee A-1, has been appointed to the important post of secretary of Subcommittee XIII on Methods of Testing.

Committee A-1 has accepted with regret the resignation of A. D. Morris of the Bayonne Bolt Corp. as chairman of Subcommittee XXVI on Bolting. Mr. Morris has served as chairman of the subcommittee since its organization in 1947. New officers appointed were J. J. Kelley, Pittsburgh Screw and Bolt Co., as chairman; A. S. Jameson, International Harvester Co., as vice-chairman; and Andrew Schwartz, Jr., Bethlehem Steel Co., as secretary.

Losses by Death.—During the year the Committee lost by death two men who had been active in its work. Howard M. Smith, Wyckoff Steel Co., had been concerned particularly with the activities of Subcommittee XV involving cold-drawn material. J. Hall Taylor, who for many years headed the Taylor Forge and Pipe Works, had been a member of Committee A-1 since 1930. Until his retirement about two years ago, he was very active in the work of Subcommittee IX and Subcommittee XXII, in the latter serving for many years as chairman of its Section on Forgings. The committee records its appreciation of the activities of these men.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

On September 26, 1950, the Administrative Committee on Standards accepted the following recommendations submitted by Committee A-1:

New Tentative Specifications for:

General Requirements for Delivery of Rolled Steel Plates of Flange and Firebox Qualities (A 20 - 50 T), and
Cold-Finished Alloy-Steel Bars (A 331–50 T).

Revision of Tentative Specifications for:

Boiler and Firebox Steel for Locomotives (A 30 - 49 T),
Carbon-Silicon Steel Plates of Intermediate Tensile Ranges for Fusion-Welded Boilers and Other Pressure Vessels (A 201 - 49 T),
Nickel-Steel Plates for Boilers and Other Pressure Vessels (A 203 - 49 T),
Molybdenum-Steel Plates for Boilers and Other Pressure Vessels (A 204 - 49 T),
High Tensile Strength Carbon-Silicon Steel Plates for Boilers and Other Pressure Vessels (Plates 6 in. and Under in Thickness) (A 212 - 49 T),
Manganese-Vanadium Steel Plates for Boilers and Other Pressure Vessels (A 225 - 49 T),
Low and Intermediate Tensile Strength Carbon-Steel Plates of Flange and Firebox Qualities (Plates 2 in. and Under in Thickness) (A 285 - 49 T),
High Tensile Strength Carbon-Manganese-Silicon Steel Plates for Boilers and Other Pressure Vessels (A 299 - 49 T),
Steel Plates for Pressure Vessels for Service at Low Temperatures (A 300 - 47 T),
Chromium-Molybdenum Steel Plates for Boilers and Other Pressure Vessels (A 301 - 49 T),
Manganese-Molybdenum Steel Plates for Boilers and Other Pressure Vessels (A 302 - 49 T),
Heat-Treated Alloy-Steel Bars (A 286 - 46 T),
Carbon-Steel Bars Subject to Mechanical Property Requirements (A 306 - 47 T), and
Hot-Rolled Alloy-Steel Bars (A 322 - 49 T).

Revision and Reversion to Tentative of Standard Specifications for:

Chromium-Manganese-Silicon (CMS) Alloy-Steel Plates for Boilers and Other Pressure Vessels (A 202 - 47), and
Open-Hearth Iron Plates of Flange Quality (A 129 - 47),

The new and revised tentatives appear in the 1950 Supplement to Book of ASTM Standards, Part 1.

On December 14, 1950, the Administrative Committee on Standards accepted the following recommendations:

Revision and Reversion to Tentative of Standard Specifications for:

Billet Steel Bars for Concrete Reinforcement (A 15 - 39),
Rail Steel Bars for Concrete Reinforcement (A 16 - 35),

Axle Steel Bars for Concrete Reinforcement (A 160 – 39), and
Minimum Requirements for the Deformation of Deformed Steel Bars for Concrete Reinforcement (A 305 – 49).

The revised specifications appear in the 1950 Supplement to Book of ASTM Standards, Part 1.

On January 25, 1951, The Administrative Committee on Standards accepted the following recommendations:

New Tentative Specifications for:

Seamless and Welded Steel Pipe for Low-Temperature Service (A 333 – 50 T).

Tentative Revision of Standard Specifications for:

Carbon- and Alloy-Steel Nuts for Bolts for High-Pressure and High-Temperature Service (A 194 – 48),
Factory-Made Wrought Carbon-Steel and Carbon-Molybdenum-Steel Welding Fittings (A 234 – 50), and
Heat-Treated Carbon-Steel Bolting Material (A 261 – 47).

Revision of Tentative Specifications for:

Alloy-Steel Bolting Materials for High-Temperature Service (A 193 – 50 T),
Alloy-Steel Bolting Materials for Low-Temperature Service (A 320 – 49 T),
Structural Steel for Ships (A 131 – 50 T),
Mild-to-Medium-Strength Carbon-Steel Castings for General Application (A 27 – 46 T),
High-Strength Steel Castings for Structural Purposes (A 148 – 46 T),
General Requirements for Delivery of Rolled Steel Plates of Flange and Firebox Qualities (A 20 – 50 T),
Open Hearth Iron Plates of Flange Quality (A 129 – 50 T),
Carbon-Silicon Steel Plates of Intermediate Tensile Ranges for Fusion-Welded Boilers and Other Pressure Vessels (A 201 – 50 T),
High Tensile Strength Carbon-Silicon Steel Plates for Boilers and Other Pressure Vessels (A 212 – 50 T),
Boiler and Firebox Steel For Locomotives (A 30 – 50 T),
Chromium-Manganese-Silicon (CMS) Alloy-Steel Plates for Boilers and Other Pressure Vessels (A 202 – 50 T),
Low and Intermediate Tensile Strength Carbon-Steel Plates of Flange and Firebox Qualities (A 285 – 50 T),
Nickel-Steel Plates for Boilers and Other Pressure Vessels (A 203 – 50 T),
Molybdenum-Steel Plates for Boilers and Other Pressure Vessels (A 204 – 50 T),
Manganese-Vanadium Steel Plates for Boilers and Other Pressure Vessels (A 225 – 50 T),
Chromium-Molybdenum Steel Plates for Boilers and Other Pressure Vessels (A 301 – 50 T),
Manganese-Molybdenum Steel Plates for Boilers and Other Pressure Vessels (A 302 – 50 T), and
Seamless Alloy-Steel Pipe for High-Temperature Service (A 158 – 50 T).

Revision and Reversion to Tentative of Standard Specifications for:

Alloy-Steel Castings for Valves, Flanges, and Fittings for High Temperature Service (A 157 – 44),
Seamless Alloy-Steel Boiler and Superheater Tubes (A 213 – 46), and
Heat Treated Steel Helical Springs (A 125 – 39).

The new and revised tentatives together with the tentative revisions of standards appear in the 1950 Supplement to Book of ASTM Standards, Part 1.

RECOMMENDATIONS AFFECTING STANDARDS

Committee A-1 is presenting for publication 4 new tentatives and is recommending the revision of 18 tentatives, 1 tentative revision, and 1 standard; the adoption as standard of 1 tentative and tentative revisions of 2 standards; the publishing of a new tentative revision of 1 standard; the revision of 17 standards with reversion to tentative; and the withdrawal of one standard.

The standards and tentatives affected, together with the revisions recommended, are given in detail in the Appendix.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

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[2] The letter ballot vote on these recommendations was favorable; the results of the ballot are on record at ASTM Headquarters.

ACTIVITIES OF SUBCOMMITTEES

The activities of the various subcommittees are so numerous that it is beyond the scope of this report to enumerate all of them. Several particularly significant activities have already been mentioned in the beginning of this report, and only the most salient will be covered in the following paragraphs.

Subcommittee VI is appointing a group to prepare a new specification for forged cylinders made by drawing or piercing methods. The same committee is engaged in revising Specifications A 273 and A 274, covering carbon and alloy steel blooms, billets, and slabs for forgings, and Specifications A 290, A 291, A 292, and A 293 covering turbine forgings. A proposed method for ultrasonic testing of forgings is also being developed.

A joint group of Subcommittees IX and XXII is studying the chemical requirements for tubular products of ferritic and austenitic stainless and heat-resisting steels with the view of setting up standard ladle and check analysis requirements, as has been done for carbon and alloy steels. As mentioned in the beginning of this report, favorable subcommittee ballots have resulted in emergency alternate provisions for the phosphorus content to a maximum of 0.045 per cent on check analysis in Specifications A 199, A 200, A 213, A 249, A 268, A 269, A 270, A 271 and A 312.[1] The section on pipe of Subcommittee XXII is appointing a group to draft a specification for fusion welded alloy steel pipe for high-temperature service of grades comparable to the present Specifications A 280, A 315, and grade T22 of Specifications A 213. If possible, these grades are to be added to present Specifications A 155. This pipe section has also appointed an eight man group to consider a specification for centrifugally cast alloy steel pipe.

Subcommittee XIII has formulated preferred definitions for yield point and yield strength when applied to pearlitic steels, and these have been forwarded to Committee E-1 for its consideration. Speed of testing has also been included in these recommendations.

A new specification for nitriding steel bar material has been under consideration in Subcommittee XV and is expected to be in final form in the near future. The Tentative Specifications for Alloy-Steel Rounds Suitable for Oil Quenching to End-Quench Hardenability Requirements (A 304) have undergone extensive revision and expansion by Subcommittee XV and are being currently balloted upon by Committee A-1. It is expected that the revised specifications will be submitted to the Administrative Committee on Standards for acceptance for publication if a favorable result is obtained.[1] Because of the current shortage of alloying elements, and the fact that only one specification (A 193) for alloy-steel bolting material is published by the Society, a new specification for quenched-and-tempered alloy-steel bolts and studs with suitable nuts has been drafted by Subcommittee XXVI and is expected to be proposed for publication during 1951. In contrast to Specification A 193, it will be designed for non-high temperature application.

Subcommittee XIX is planning on forming a group to study and prepare a specification for commercial quality cold-rolled sheets.

Expected to be approved for publication during 1951 is a specification comprising eight grades of ferritic and austenitic steel castings for high-temperature service. Also under consideration in the section on welding fittings of Subcommittee XXII is a specification for austenitic steel welding fittings.

A special group is attempting a correlation of the grade symbol numbering system in ASTM steel specifications where identical or similar material may be included in different product forms.

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[1] See editorial note, p. 89.

This report has been submitted to letter ballot of the committee, which consists of 272 members; 207 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

N. L. MOCHEL,
Chairman.

J. S. WORTH,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, the following Emergency Alternate Provisions, submitted by Committee A-1, were accepted by the Administrative Committee on Standards on the date indicated:

Designation	Emergency Alternate Provisions In	Date Issued
	Standard Specifications for:	
EA - A 26	Steel Tires (A 26 - 39)	Aug. 21, 1951
*EA - A 268	Seamless and Welded Ferritic Stainless Steel Tubing for General Service (A 268 - 47)	July 10, 1951
*EA - A 269	Seamless and Welded Austenitic Stainless Steel Tubing for General Service (A 269 - 47)	July 10, 1951 and Oct. 18, 1951
*EA - A 270	Seamless and Welded Austenitic Stainless Steel Sanitary Tubing (A 270 - 50)	July 10, 1951
*EA - A 271	Seamless Austenitic Chromium-Nickel Steel Still Tubes for Refinery Service (A 271 - 47)	July 10, 1951 and Oct. 18, 1951
	Tentative Specifications for:	
EA - A 199	Seamless Cold-Drawn Intermediate Alloy-Steel Heat-Exchanger and Condenser Tubes (A 199 - 51 T)	July 10, 1951
EA - A 200	Seamless Intermediate Alloy-Steel Still Tubes for Refinery Service (A 200 - 51 T)	July 10, 1951
EA - A 213	Seamless Alloy-Steel Boiler, Superheater, and Heat-Exchanger Tubes (A 213 - 51 T)	July 10, 1951
EA - A 249	Welded Austenitic Stainless Steel Boiler, Superheater, Heat Exchanger and Condenser Tubes (A 249 - 51 T)	July 10, 1951
*EA - A 312	Seamless and Welded Austenitic Stainless Steel Pipe (A 312 - 51 T)	July 10, 1951 and Oct. 18, 1951
EA - A 329	Heat-Treated Steel Tires (A 329 - 50 T)	Aug. 21, 1951

* Jointly with Committee A-10.

These emergency provisions have been published in the ASTM BULLETIN and have also been issued in the form of pink stickers for attachment to the standards to which they apply.

On September 12, 1951, the following recommendations submitted by Committee A-1 were accepted by the Administrative Committee on Standards:

Revision of Tentative Specifications for:

Alloy-Steel Rounds Suitable for Oil Quenching to End-Quench Hardenability Requirements (A 304 - 47 T),
Quenched and Tempered Steel Bolts and Studs with Suitable Nuts and Plain Washers (A 325 - 49 T),
Welded and Seamless Steel Pipe (A 53 - 51 T), and
Seamless Alloy Steel Boiler and Superheater Tubes (A 213 - 51 T).

Revision and Reversion to Tentative of Standard Specifications for:

Structural Nickel Steel (A 8 - 46),
Welded Alloyed Open-Hearth Iron Pipe (A 253 - 47),
Welded Alloy-Steel Boiler and Superheater Tubes (A 249 - 47),
Electric-Fusion Welded Steel Pipe (Sizes 4 in. to but Not Including 30 in.) (A 139 - 46), and
Electric-Fusion Welded Steel Pipe (Sizes 30 in. and Over) (A 134 - 42).

The revised specifications appear in the 1951 Supplement to Book of ASTM Standards, Part 1.

The following recommendations, submitted by Committee A-1, jointly with Committee A-10 on Iron-Chromium, Iron-Chromium-Nickel and Related Alloys, were accepted by the Administrative Committee on Standards on October 29, 1951, and appear in the 1951 Supplement to Book of ASTM Standards, Part 1:

Revision of Tentative Specifications for:
Seamless and Welded Austenitic Steel Pipe (A 312 - 51 T).

Tentative Revision of Standard Specifications for:
Seamless Austenitic Chromium-Nickel Steel Still Tubes for Refinery Service (A 271 - 47).

APPENDIX

RECOMMENDATIONS AFFECTING STANDARDS ON STEEL

In this Appendix recommendations are given affecting certain specifications, both standard and tentative, covering various steel products. These specifications appear in their present form in either the 1949 Book of ASTM Standards, Part 1, or in the 1950 Supplement, Part 1, as indicated by the final number in the ASTM designation.

Committee A-1 recommends that the following definition of heat-treated lots be included in tubular specifications under its jurisdiction:

The term "lot" applies to all tubes (or pipes) of the same nominal size and wall thickness (or schedule) which are produced from the same heat of steel and subjected to the same finishing treatment in a continuous furnace; when final heat treatment is in a batch type furnace, the lot shall include only those tubes (or pipes) which are heat treated in the same furnace charge. When no heat treatment is performed following final forming operations, the lot shall include hot rolled material only or cold drawn material only.

Since there will be specifications where specific restrictions on the size of lots may already be imposed, it is recommended that the above definition be accepted with the provision that a certain amount of editorial work will be necessary for the application of this definition to individual specifications.

NEW TENTATIVES

The committee recommends the following four new tentatives, as appended hereto,[1] be accepted for publication as tentative:

Tentative Specifications for Nickel-Chromium-Molybdenum Steel Bars for Springs:

These specifications have been under consideration for approximately three years. It may be noted that these proposed specifications include particular grades of steels specified to hardenability requirements.

Tentative Specification for Alloy Steel Seamless Drum Forgings:

This specification includes 14 grades of ferritic and austenitic steels suitable for alloy steel drum forgings. It will be a companion to the present Tentative Specification for Carbon-Steel Seamless Drum Forgings (A 266 – 49 T).

Tentative Specifications for Seamless and Welded Steel Tubes for Low Temperature Service:

Three grades of ferritic steel subject to impact test requirements are included in these specifications, which have been under consideration for a number of years.

Tentative Specifications for Seamless Ferritic Alloy Steel Pipe for High Temperature Service:

These specifications represent a consolidation in one document of various ferritic alloy grades of seamless steel pipe covered in Specifications A 158, A 206,

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[1] These new tentatives were accepted by the Society and appear in the 1951 Supplement to Book of ASTM Standard. Part 1.

A 280, and A 315 with the addition of two new grades.

REVISIONS OF TENTATIVES

The committee recommends revisions in the following 18 tentatives:

Tentative Specification for General Requirements for Delivery of Rolled Steel Plates, Shapes, Sheet Piling, and Bars for Structural Use (A 6 – 50 T):

Section 8(c).—In the second sentence delete the words "and which do not affect the full utility of the pieces" following the word "depth."

TABLE I.—CHEMICAL REQUIREMENTS.

	Grade A		Grade B	
	Ladle Analysis	Check Analysis	Ladle Analysis	Check Analysis
Carbon, max, per cent	...	...	0.28	0.31
Manganese, per cent	0.30 to 0.60	0.27 to 0.63	0.30 to 0.80	0.27 to 0.83
Phosphorus, max, per cent	0.040	0.048	0.040	0.048
Sulfur, max, per cent	0.050	0.058	0.050	0.058

Tentative Specifications for Boiler Rivet Steel and Rivets (A 31 – 49 T):

Section 3.—Revise to read "The steel shall conform to the requirements as to chemical composition prescribed in Table I."

New Table.—Add a Table I to read as shown in the accompanying Table I, renumbering subsequent tables accordingly.

Section 4.—In the first sentence delete the words "and silicon for grade B."

Revise the last portion of the third sentence to read "and shall conform to the requirements for ladle analysis prescribed in Table I."

Section 5.—Revise the second sentence to read "The chemical composition thus determined shall conform to the requirements for check analysis specified in Table I."

Section 7(b).—Delete.

Section 9(a).—Delete reference to quench bend tests.

Section 17(a).—Delete the last line of the table in this paragraph covering rivets over 1½ to 2 in., inclusive, in diameter.

Tentative Specifications for Seamless Carbon-Steel Pipe for High-Temperature Service (A 106 – 48 T):

Table I.—Revise to read as shown in the accompanying Table II.

Tentative Specifications for Seamless Alloy-Steel Pipe for High-Temperature Service (A 158 – 50 T):

Table I.—Revise the chemical requirements for grades P3a, P3b, P11 and P15 to read as shown in the accompanying Table III.

Tentative Specifications for Heat-Treated Carbon- and Alloy-Steel Track Bolts and Nuts (A 183 – 49 T):

Section 1.—Revise to read "These specifications cover heat treated carbon and alloy steel track bolts; also carbon steel nuts."

Section 3.—In Paragraph (*b*) revise the reference to "sulfurized nut stock grades" to read "free-cutting grades."

Revise Paragraph (*c*) to read "When medium carbon nuts are specified, the steel shall contain a carbon content of 0.40 to 0.55 per cent."

Section 4.—Revise Paragraphs (*a*) and (*b*) to read as follows:

4. (*a*) A carbon determination shall be made of each melt of bessemer steel, and determinations for manganese, phosphorus, and sulfur representing the average of the melts applied for each 8-hr period.

(*b*) An analysis of each heat of open-hearth or electric-furnace steel shall be made to determine the percentage of carbon, manganese, phosphorus, and sulfur; also any alloy element when used.

TABLE II.—CHEMICAL REQUIREMENTS.

	Grade A		Grade B	
	Ladle Analysis	Check Analysis	Ladle Analysis	Check Analysis
Carbon, max, per cent.........	0.23	0.25	0.27	0.30
Manganese, per cent...........	0.30 to 0.90	0.27 to 0.93	0.35 to 1.00	0.29 to 1.06
Phosphorus, max, per cent......	0.040	0.048	0.040	0.048
Sulfur, max, per cent..........	0.050	0.058	0.050	0.058
Silicon, min, per cent..........	0.12	0.10	0.12	0.10

TABLE III.—CHEMICAL REQUIREMENTS.

	Grade P3a		Grade P3b		Grade P11		Grade P15	
	Ladle Analysis	Check Analysis	Ladle Analysis	Check Analysis	Ladle Analysis	Check Analysis	Ladle Analysis	Check Analysis
Carbon, max, per cent.....	0.14	0.15	0.14	0.15	0.14	0.15	0.14	0.15
Manganese, per cent.....	0.33 to 0.57	0.30 to 0.60	0.33 to 0.57	0.30 to 0.60	0.33 to 0.57	0.30 to 0.60	0.33 to 0.57	0.30 to 0.60
Phosphorus, max, per cent	0.025	0.030	0.025	0.030	0.025	0.030	0.025	0.030
Sulfur, max, per cent.....	0.025	0.030	0.025	0.030	0.025	0.030	0.025	0.030
Silicon, per cent	0.45 to 0.75	0.40 to 0.80	0.45 max	0.50 max	0.55 to 0.95	0.50 to 1.00	1.20 to 1.60	1.15 to 1.65
Chromium, per cent.....	1.50 to 2.00	1.45 to 2.05	1.75 to 2.25	1.65 to 2.35	1.05 to 1.45	1.00 to 1.50	...	...
Molybdenum, per cent.....	0.63 to 0.78	0.60 to 0.81	0.48 to 0.63	0.45 to 0.66	0.48 to 0.63	0.45 to 0.66	0.48 to 0.63	0.45 to 0.66

In Paragraph (*c*) in the first sentence change the words "the heats or blows" to read "each heat or melt."

Section 5.—In the first sentence change the word "blow" to "melt." In the second sentence change the reference to "Section 3(*a*)" to read "Section 3."

Section 6.—Revise to read as follows:

6. (*a*) *Tension Tests.*—Tension test specimens shall be taken from the finished bolts and shall conform to the following minimum requirements as to tensile properties:

	Carbon Steel	Alloy Steel
Tensile strength, psi......	110 000	115 000
Yield point, psi..........	80 000	85 000
Elongation in 2 in., per cent..................	12	15
Reduction of area, per cent..................	25	40

(*b*) The yield point shall be determined by the drop of the beam or halt in the gage of the testing machine operated at a crosshead speed not in excess of $\frac{1}{8}$ in. per min. The tensile strength shall be determined at a speed not in excess of $1\frac{1}{2}$ in. per min.

(*c*) The tension test specimen taken from the finished bolt shall conform to the dimensions shown in Fig. 1 (retain present Fig. 1).

Section 7.—Delete, renumbering subsequent sections accordingly.

Section 8.—Revise the section title to read "Bend Tests." In Paragraph (*a*) in the first sentence delete the words "grades 1 and 2."

Section 9.—Revise to read as follows:

9. The threads of the nut shall not "strip" or the bolt break when the bolt, with the nut fully mounted, is tested in tension to the following loads for the diameter and grade ordered:

Nominal Bolt Diameter, in.	Stress Area,[a] sq in.	Minimum Load, lb	
		Carbon Steel, 110 000 psi	Alloy Steel 115 000 psi
3/4	0.3340	36 740	38 410
13/16	0.4011	44 120	46 130
7/8	0.4612	50 730	53 040
15/16	0.5395	59 340	62 040
1	0.6051	66 560	69 590
1 1/16	0.6942	76 360	79 830
1 1/8	0.7627	83 900	87 710
1 1/4	0.9684	106 520	111 370

[a] The stress area is the assumed area of a circle having a diameter equal to the average of the mean pitch and minor diameters with class 3 tolerances shown on page 16 of the American Standard for Unified and American Screw Threads (A.S.A. No. B1.1–1949). The above table is predicated on a minimum nut thickness equal to the nominal diameter of the bolt.

Table II.—Delete.

Section 11.—Revise to read as follows:

11. The bolts and nuts shall conform to the plans specified by the purchaser, subject to the following variations:

(*a*) The nominal size of bolts shall be the over-all diameter of the threads.

(*b*) The outside diameter of the finished rolled threads shall not exceed the diameter of the shank of a cut thread bolt of corresponding size.

The diameter of the rolled threads shall not exceed the diameter of the shank by more than 1/16 in. for bolts 7/8 in. and under in diameter, nor more than 3/32 in. for bolts 1 in. and over in diameter.

Shank diameter for cut thread bolt	+1/64 in. or −1/32 in.
Neck dimensions	+1/32 in. or −1/32 in.
Length under head	+1/8 in. or −1/8 in.
Height and diameter of head	+1/16 in. or −1/16 in.
Nut width	−(0.05 × thread diameter of bolt) in.
Nut height	+ or −(0.016 × thread diameter of bolt +0. 12) in.

Section 13.—In the first sentence change the work "nicks" to read "nicking." In the second sentence delete the words "and firmly joined to."

Sections 14 and 15.—Renumber as Sections 13 and 14 and revise to read as follows:

13. The threads on bolts may be rolled or cut. The threads on bolts and nuts shall conform to the American National Form of Thread as shown in the American Standard for Unified and American Screw Threads (A.S.A. No. B1.1–1949) or any later revision thereof. A free fit shall be furnished unless otherwise specified by the purchaser in accordance with the following provisions:

(*a*) *Free Fit.*—The threads of the bolts and nuts shall conform as nearly as practicable to the American Standard limits for Coarse Thread Series Free Fit (class 2) screws and nuts. The maximum tightness shall be 5-lb pull applied to the end of a 24-in. wrench.

(*b*) *Wrench Turn Fit.*—The nut shall have a free fit for at least two threads in starting on the bolt and when fully engaged and for the remainder of the screw length shall show the following minimum and maximum resistance to turning as expressed by pounds pull applied to the end of a 24-in. wrench.

	Minimum, lb	Maximum, lb
Low carbon nuts (Section 3 (*b*))	5	45
Medium carbon nuts (Section 3 (*c*))	5	55

Before packing, nuts shall be screwed on the bolts enough turns to hold them in place until used.

14. (*a*) A letter or brand indicating the manufacturer, together with the initial "A" if alloy bolts, shall be pressed on the head of the bolt when it is formed.

(*b*) All containers shall be marked by the manufacturer as follows:

(*1*) Name of manufacturer,
(*2*) Material (carbon or alloy),
(*3*) Size of bolt (diameter and length), and
(*4*) Weight.

Section 16.—Change the word "material" to read "bolts and nuts" in the first and second sentences.

Section 17.—In Paragraph (*a*) delete the words "by the purchaser." In Paragraph (*b*) change the word "material" to read "bolts and nuts."

Section 18.—In the first sentence change the word "material" to read "bolts."

Tentative Specifications for Alloy-Steel Bolting Materials for High-Temperature Service (A 193 – 50a T):

Section 1(b).—Revise the first sentence to read "Thirteen grades are covered, including nine ferritic steels and four austenitic steels designated BA, B5, B6, etc."

Table I.—Delete the present grade B8 and replace with three grades as shown in the accompanying Table IV, retaining the present footnotes.

Table II.—In the first column under austenitic steels revise to read "B8, B8c, B8t and B8F."

Section 10(d).—Delete.

TABLE IV.—CHEMICAL REQUIREMENTS.

Identification Symbol	B8		B8c		B8t	
Grade	Unstabilized 18 Chromium-8 Nickel (A.I.S.I. Type 304)		Stabilized 18 Chromium-8 Nickel (A.I.S.I. Type 347)		Stabilized 18 Chromium-8 Nickel (A.I.S.I. Type 321)	
	Range	Check Variation, Over or Under[a]	Range	Check Variation, Over or Under[a]	Range	Check Variation, Over or Under[a]
Carbon, per cent	0.08 max	0.01 over	0.08 max	0.01 over	0.08 max	0.01 over
Manganese, per cent	2.00 max	0.04 over	2.00 max	0.04 over	2.00 max	0.04 over
Phosphorus, per cent	0 04 max	0.005 over	0.04 max	0.005 over	0.04 max	0.005 over
Sulfur, per cent	0.03 max	0.005 over	0.03 max	0.005 over	0.03 max	0.005 over
Silicon, per cent	1.00 max	0.05 over	1.00 max	0.05 over	1.00 max	0.05 over
Nickel, per cent	8.00 to 11.00	0.15	9.00 to 12.00	0.15	8.00 to 11.00	0 15
Chromium, per cent	18.00 to 20.00	0 20	17.00 to 19.00	0.20	17.00 to 19.00	0.20
Titanium, per cent	....	....	....	....	5X carbon content, min	0.05 over
Columbium, per cent	....	....	10X carbon content, min	0.05	....	....

Tentative Specifications for Nickel-Steel Plates for Boilers and Other Pressure Vessels (A 203 - 50 T):

Table II.—For grade C revise the tensile strength range from the present "75,000 to 87,000" to read "75,000 to 90,000" psi.

Section 6(b).—Revise the last phrase to read "nor 93,000 psi for grade C."

Tentative Specifications for Seamless Carbon-Molybdenum Alloy-Steel Pipe for High Temperature Service (A 206 - 48 T):

Section 5.—Revise the table of chemical requirements for grade P1 in this section to read as follows:

	Ladle Analysis	Check Analysis
Carbon, per cent	0.11 to 0.19	0.10 to 0.20
Manganese, per cent	0.33 to 0.77	0.30 to 0.80
Phosphorus, max, per cent	0.040	0.045
Sulfur, max, per cent	0.040	0.045
Silicon, per cent	0.15 to 0.45	0.10 to 0.50
Molybdenum, per cent	0.48 to 0.63	0.45 to 0.66

Tentative Specifications for Seamless Alloy-Steel Boiler and Superheater Tubes (A 213 - 50 T):

Table I.—Revise the chemical requirements for grades T3, T11, T12, T14, T17, T21 and T22 to read as shown in the accompanying Table V.

Tentative Specification for Carbon and Alloy Steel Ring and Disk Forgings (A 243 - 49 T):

Section 12(g).—Revise to read as follows:

(*g*) Unless otherwise specified, the axis of the specimen shall be located at any point midway between the parallel surfaces of the test extension if added to the periphery or shall be located at any point midway between the center and surface of solid forgings and at any point midway between the inner and outer surfaces of the wall of a hollow or bored forging. The axis of the specimen shall be perpendicular to the axis of the forging, and also perpendicular to the radius of the forging that intercepts its midlength point. However, where the length of the cover or head is half the diameter or greater, the test specimen shall be located at mid-wall or mid-radius as described above except that the axis of the specimen may be parallel to the axis of the forging.

Tentative Specification for Carbon Steel Seamless Drum Forgings (A 266 – 49 T):

Section 6(a).—Revise to read "Forged pressure vessels for steam power service shall have the inner surface machined. Unfired pressure vessels shall have the inner surfaces sufficiently freed from scale to permit inspection, unless otherwise specified."

TABLE V.—CHEMICAL REQUIREMENTS OF FERRITIC STEEL.

Identification Symbol	T3		T11		T12		T14	
Grade	Chromium-Molybdenum		Chromium-Silicon-Molybdenum		Chromium-Molybdenum		Chromium-Molybdenum	
	Ladle Analysis	Check Analysis	Ladle Analysis	Check Analysis	Ladle Analysis	Check Analysis	Ladle Analysis	Check Analysis
Carbon, max, per cent	0.14	0.15	0.14	0.15	0.14	0.15	0.14	0.15
Manganese, per cent	0.33 to 0.57	0.30 to 0.60	0.33 to 0.57	0.30 to 0.60	0.33 to 0.58	0.30 to 0.61	0.33 to 0.57	0.30 to 0.60
Phosphorus, max, per cent	0.025	0.030	0.025	0.030	0.040	0.045	0.025	0.030
Sulfur, max, per cent	0.025	0.030	0.025	0.030	0.040	0.045	0.025	0.030
Silicon, per cent	0.45 to 0.75	0.40 to 0.80	0.55 to 0.95	0.50 to 1.00	0.28 max	0.30 max	0.45 max	0.50 max
Chromium, per cent	1.50 to 2.00	1.45 to 2.05	1.05 to 1.45	1.00 to 1.50	0.85 to 1.20	0.80 to 1.25	1.75 to 2.25	1.65 to 2.35
Molybdenum, per cent	0.63 to 0.78	0.60 to 0.81	0.48 to 0.63	0.45 to 0.66	0.48 to 0.63	0.45 to 0.66	0.48 to 0.63	0.45 to 0.66

Identification Symbol	T17		T21		T22	
Grade	Chromium-Vanadium		Chromium-Molybdenum		Chromium-Molybdenum	
	Ladle Analysis	Check Analysis	Ladle Analysis	Check Analysis	Ladle Analysis	Check Analysis
Carbon, per cent	0.16 to 0.24	0.15 to 0.25	0.14 max	0.15 max	0.14 max	0.15 max
Manganese, per cent	0.33 to 0.58	0.30 to 0.61	0.33 to 0.57	0.30 to 0.60	0.33 to 0.57	0.30 to 0.60
Phosphorus, max, per cent	0.040	0.045	0.025	0.030	0.025	0.030
Sulfur, max, per cent	0.040	0.045	0.025	0.030	0.025	0.030
Silicon, per cent	0.17 to 0.33	0.15 to 0.35	0.45 max	0.50 max	0.45 max	0.50 max
Chromium, per cent	0.85 to 1.20	0.80 to 1.25	2.75 to 3.25	2.65 to 3.35	2.00 to 2.50	1.90 to 2.60
Molybdenum, per cent	....	....	0.83 to 1.03	0.80 to 1.06	0.90 to 1.10	0.87 to 1.13
Vanadium, min, per cent	0.17	0.15	....	....	....	....

Section 13(c).—Revise to read as follows:

(*c*) One tension test specimen shall be taken to represent each single or multiple cover or head forging. Unless otherwise specified, the axis of the specimen shall be located at any point midway between the parallel surfaces of the test extension if added to the periphery or shall be located at any point midway between the center and surface of solid forgings and at any point midway between the inner and outer surfaces of the wall of a hollow or bored forging. The axis of the specimen shall be perpendicular to the radius of the forging that intercepts its mid-length point. However, where the length of the cover or head is half the diameter or greater, the test specimen shall be located at mid-wall or mid-radius as described above except that the axis of the specimen may be parallel to the axis of the forging.

Tentative Specifications for Seamless Chromium-Molybdenum Alloy-Steel Pipe for Service at High Temperatures (A 280 – 48 T):

Section 5.—Revise the table of chemical requirements to read as follows:

	Ladle Analysis	Check Analysis
Carbon, per cent	0.11 to 0.19	0.10 to 0.20
Manganese, per cent	0.33 to 0.58	0.30 to 0.61
Phosphorus, max, per cent	0.040	0.045
Sulfur, max, per cent	0.040	0.045
Silicon, per cent	0.12 to 0.28	0.10 to 0.30
Molybdenum, per cent	0.48 to 0.63	0.45 to 0.66
Chromium, per cent	0.53 to 0.78	0.50 to 0.81

Tentative Specification for Low and Intermediate Tensile Strength Carbon-Steel Plates of Structural Quality (Plates 2 in. and Under in Thickness) (A 284 – 50 T):

Section 1.—Revise to read as follows:

1. (*a*) This specification covers carbon steel plates subject to mechanical test requirements and intended for machine parts and general construction (other than pressure vessels) by gas cutting, welding, or other means.

(*b*) A definite silicon content is specified in order to limit the carbon to the lowest practicable amount consistent with the specified tensile strength and the thickness of the material. Welding technique is of fundamental importance and it is presupposed that welding procedure will be in accordance with approved methods.

(*c*) The maximum thickness for plates to be delivered under this specification is 12 in. for plates of grades A, B and C, and 8 in. for plates of grade D.

Table I.—Revise to read as shown in the accompanying Table VI.

Table II.—Add tensile requirements for grade D as follows:

Tensile strength, min, psi	60 000
Yield point, min, psi	33 000
Elongation in 8 in., min, per cent	21
Elongation in 2 in., min, per cent	22

Table III.—Add bend test requirements for grade D as follows:

Thickness of Material, in.	Ratio of Bend Diameter to Thickness of Specimen
¾ and under	½
Over ¾ to 1, incl	1
Over 1 to 1½, incl	1½
Over 1½ to 2, incl	2½
Over 2 to 3, incl	3
Over 3 to 4, incl	3½
Over 4	4

Tentative Specification for Chromium-Molybdenum Steel Plates for Boilers and Other Pressure Vessels (A 301 – 50 T):

Section 1.—In the first sentence after the word "plates" add "of two compositions, designated grades A and B."

Section 3.—In Paragraph (*b*) revise to read "The steel for grade A shall be a coarse grained. . . ."

Add a new Paragraph (*c*) to read "Specific limitations on deoxidation practice or grain size requirements for the steel for grade B shall be a matter for agreement between the manufacturer and the purchaser."

TABLE VI.—CHEMICAL REQUIREMENTS.

	Grade A		Grade B		Grade C		Grade D	
	Ladle Analysis	Check Analysis	Ladle Analysis	Check Analysis	Ladle Analysis	Check Analysis	Ladle Analysis	Check Analysis
Carbon, max, per cent for thickness as follows:								
1 in. and under	0.20	0.24	0.22	0.26	0.25	0.29	0.29	0.33
Over 1 to 2 in., incl	0.22	0.26	0.25	0.29	0.28	0.32	0.32	0.36
Over 2 to 4 in., incl	0.25	0.29	0.28	0.32	0.32	0.36	0.35	0.39
Over 4 to 8 in., incl	0.29	0.33	0.32	0.36	0.36	0.40	0.38	0.42
Over 8 to 12 in., incl	0.32	0.36	0.35	0.39	0.38	0.42	...	...
Manganese, max, per cent	0.90	0.94	0.90	.94	0.90	0.94	0.90	0.94
Phosphorus, max, per cent	0.04	0.05	0.04	0.05	0.04	0.05	0.04	0.05
Sulfur, max, per cent	0.05	0.063	0.05	0.063	0.05	0.063	0.05	0.063
Silicon per cent	0.10 to 0.30	0.08 to 0.33	0.15 to 0.30	0.13 to 0.33	0.15 to 0.30	0.13 to 0.33	0.15 to 0.30	0.13 to 0.33

Section 4.—In Paragraph (*a*) revise the first sentence to read "All plates shall be treated to produce grain refinement either. . . ."

Delete the present Paragraph (*e*).

Table I.—Add chemical requirements for grade B as follows:

Carbon, max, per cent	0.21
Manganese, max, per cent	0.65
Phosphorus, max, per cent:	
Flange	0.040
Firebox	0.035
Sulfur, max, per cent:	
Flange	0.050
Firebox	0.040
Silicon, per cent	0.15 to 0.30
Chromium, per cent	0.80 to 1.10
Molybdenum, per cent	0.45 to 0.65

Table II.—Add tensile strength requirements for grade B as follows, retaining the present footnotes *a* and *b*:

Tensile strength, psi	60 000 to 77 000
Yield point, min, psi	0.55 tens str
but in no case less than	35 000
Elongation in 8 in., min, per cent:	
Flange	$\frac{1\,600\,000^{a}}{\text{tens str}}$
Firebox	$\frac{1\,650\,000^{a}}{\text{tens str}}$
Elongation in 2 in., min, per cent:	
Firebox	$\frac{1\,750\,000^{b}}{\text{tens str}}$

Section 6.—In Paragraph (*b*) revise the last portion to read "85,000 psi for grade A and 80,000 psi for grade B."

In Paragraph (*e*) revise to read "For plates over $\frac{1}{4}$ to $\frac{3}{4}$ in. in thickness, if the. . . ."

Section 8.—Delete Paragraph (*b*).

Section 9.—Omit reference to the homogeneity test.

Section 10.—Revise the letters "Cr-Mo" to read "Cr-Mo-A" or "Cr-Mo-B."

Tentative Specifications for Seamless and Welded Austenitic Stainless Steel Pipe (A 312 – 48 T):

Section 1.—In Paragraph (*b*) revise the second sentence to read "Seven grades are covered."

Add a new Paragraph (*d*) to read "Optional supplementary requirements are provided for pipe where a greater degree of examination is desired. These supplementary requirements call for additional tests to be made at the expense of the purchaser, and when desired, one or more of these may be specified in the order."

Section 3(*a*).—Revise to read "The pipe shall be made by the seamless or an automatic welding process, with no addition of filler metal in the welding operation."

Table I.—Add two new grades with chemical requirements as follows:

Identification Symbol	TP 309	TP 310
Grade	Chromium-Nickel	Chromium-Nickel
Carbon, max, per cent	0.15	0.15
Manganese, max, per cent	2.00	2.00
Phosphorus, max, per cent	0.030	0.030
Sulfur, max, per cent	0.030	0.030
Silicon, max, per cent	0.75	0.75
Nickel, per cent	12.0 to 15.0	19.0 to 22.0
Chromium, per cent	22.0 to 24.0	24.0 to 26.0

Supplementary Requirements.—At the end of the specifications add a section titled "Supplementary Requirements for Pipe Requiring Special Consideration" to include the following:

S1. These requirements shall not be considered unless specified in the order, in which event the supplementary tests specified in Paragraphs S2 to S5 shall be made at the mill, unless otherwise agreed upon, at the purchaser's expense and witnessed by his inspector before shipment of the material.

S2. *Check Analysis.*—Check analysis may be made on any length of pipe. Individual lengths failing to conform to the chemical requirements specified in Section 6 shall be rejected.

S3. *Transverse Tension Tests.*—Transverse tension tests may be made on specimens from both ends of each length of pipe. This requirements applies only to pipe 8 in. and over in nominal diameter. If the specimen from either end of any length fails to conform to the mechanical properties specified in Section 9, that length shall be rejected.

S4. *Flattening Test.*—The flattening test specified in Section 11 may be made on specimens from both ends of each length of pipe. This requirement applies only to pipe over 2 in. in nominal diameter. Crop ends may be used. If the specimen from either end of any length fails to conform to the specified requirements, that length shall be rejected.

S5. *Etching Tests.*—The steel shall have a homogeneous structure as shown by an etching test. Etching tests* may be made on sections from any pipe, and shall show sound and reasonably uniform material, free from injurious laminations, cracks, and similar objectionable defects. If the specimen from any length shows objectionable defects, that length may be rejected.

Appendix I.—Add a new schedule 5S as follows:

Nominal Pipe Size, in.	Nominal Wall Thickness, in.
½	0.065
¾	0.065
1	0.065
1¼	0.065
1½	0.065
2	0.065
2½	0.083
3	0.083
3½	0.083
4	0.083
5	0.109
6	0.109
8	0.109
10	0.134
12	0.165

Tentative Specifications for Seamless 1 Per Cent Chromium, 0.5 Per Cent Molybdenum Alloy-Steel Pipe for Service at High Temperatures (A 315 – 48 T):

Section 2(d).—Revise to read "The steel shall be made by coarse grain melting practice. Specific limitations, if any, on grain size or deoxidation practice shall be a matter of agreement between the manufacturer and the purchaser."

Section 5.—Revise the table of chemical requirements to read as follows:

	Ladle Analysis	Check Analysis
Carbon, max, per cent	0.14	0.15
Manganese, per cent	0.33 to 0.58	0.30 to 0.61
Phosphorus, max, per cent	0.040	0.045
Sulfur, max, per cent	0.040	0.045
Silicon, max, per cent	0.28	0.30
Molybdenum, per cent	0.48 to 0.63	0.45 to 0.66
Chromium, per cent	0.85 to 1.20	0.80 to 1.25

Tentative Specifications for Alloy-Steel Bolting Materials for Low-Temperature Service (A 320 – 50 T):

Section 1(b).—Revise the first sentence to read "Seven grades are covered, including both. . . ."

Section 4(a).—Revise the last sentence to read "Grades L8, L8c, L8t and L8F shall be annealed by. . . ."

Table I.—Delete the present grade L8 and replace with three grades L8, L8c and L8t whose chemical requirements are the same as grades B8, B8c and B8t shown in the accompanying Table IV.

Table II.—In the first column change the second and third lines to read "L8, L8c, L8t and L8F, annealed" and "L8, L8c, L8t and L8F, cold drawn," respectively.

Table III.—Add grades L8c and L8t with a highest acceptance test temperature of −300 F.

Section 11(d).—Delete.

Section 14.—Revise the first sentence of Paragraph (*b*) to read "Bolts, studs, and stud bolts of grades L8, L8c, L8t and L8F shall be equipped with austenitic alloy nuts conforming to grades 8, 8c, 8t and 8F of ASTM Specifications A 194."

In Paragraph (*c*) revise the last sentence to read "If the purchaser requires nuts with a Charpy impact strength of not less than 15 ft-lb at temperatures below −150 F, he may require that the nuts conform to grades 8, 8c, 8t or 8F of ASTM Specifications A 194."

Tentative Methods and Definitions for Mechanical Testing of Steel Bars (A 330 – 50 T):

Section 9.—Revise to read as follows:

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* Pending development of etching methods applicable to the product covered by these specifications, it is recommended that the Recommended Practice for a Standard Macro Etch Test for Routine Inspection of Iron and Steel, described in the *Metals Handbook* Am. Soc. for Metals, 1948 edition, p. 389, be followed.

9. Speed of testing may be defined in terms of relative movement of the heads of the testing machine, in terms of rate of stressing the specimen or in terms of rate of straining the specimen. For most carbon steels, specifying the rate of relative movement of the heads of "crosshead speed" is adequate. In any case, the speed of testing should not be greater than that at which load and other readings can be made accurately. The following limitations on crosshead speed are recommended:

(*a*) From the start of loading the specimen until a value estimated as one-half of the yield point is reached, any convenient speed of the crosshead may be used.

(*b*) When the value estimated as being one-half of the yield point is reached, the speed of the crosshead under load shall be adjusted so that its rate of travel will be approximately, but not in excess of $\frac{1}{16}$ in. per min per inch of gage length. This speed shall be maintained through the yield point.

(*c*) If the machine is equipped with a device to indicate the rate of loading, the speed of the machine from one-half the estimated yield point or yield strength to the yield point or yield strength may be adjusted so that the rate of loading does not exceed 100,000 psi per minute. The choice of the above rate of loading limitation, instead of a crosshead speed limitation, shall be by mutual agreement between the purchaser and the manufacturer when proper testing equipment is available.

(*d*) In determining the ultimate tensile strength the crosshead speed under load shall not exceed $\frac{1}{2}$ in. per min per inch of gage length.

(*e*) It is permissible to set the crosshead speed by adjusting the free-running speed to the specified values, inasmuch as the speed under load at such machine setting will then be less than the specified values.

Section 12(*a*).—Revise Item (*2*) to read as follows:

(*2*) *Total Strain Method Using Dividers.*—In the divider method the observer, with a pair of dividers or other suitable apparatus, watches for visible elongation between two gage marks on the specimen. When visible stretch is observed, the load at that instant is noted, and the stress corresponding to the load is taken as the yield point.

TENTATIVE REVISION OF STANDARDS

The committee recommends a revision of two standards for publication as tentative as indicated below:

Standard Specifications for Oil-Tempered Steel Spring Wire (A 229 – 41):

Table I.—Increase all minimum tensile strength values by 5000 psi and delete footnote *a*.

The committee also recommends a revision in the existing tentative revision on one standard as follows:

Standard Specifications for Heat-Treated Carbon-Steel Bolting Material (A 261 – 47):

Section 12.—Delete the present Paragraph (*d*) in the tentative revision of this section issued January 25, 1951.

ADOPTION OF TENTATIVE AS STANDARD

Tentative Specifications for Heat-Treated Steel Leaf Springs (A 147 – 49 T):

The committee recommends that these specifications be approved for reference to letter ballot of the Society for adoption as standard, to replace the Standard Specifications for Heat-Treated Steel Elliptical Springs (A 147 – 39), which are recommended for withdrawal.

ADOPTION AS STANDARD OF TENTATIVE REVISIONS OF STANDARDS

Standard Specifications for Steel Music Spring Wire (A 228 – 41):

The committee recommends that the present tentative revision of Table I be adopted as standard and incorporated in these specifications.

Standard Specifications for Carbon- and Alloy-Steel Nuts for Bolts for High-Pressure and High-Temperature Service (A 194 – 48):

The committee recommends that the present tentative revisions be adopted as standard and incorporated in these specifications, with revision as indicated below:

Section 1(b).—Revise the fifth line to read "Grades 3, 4, 8, 8c, 8t and 8F for service under the most severe conditions."

Section 3.—Add Paragraphs (*d*) and (*e*) to read as follows:

> (*d*) Grades 8, 8c, 8t and 8F nuts shall be made by the hot forged or cold punching process or machined from forged or rolled bars and should be annealed by quenching in water from a minimum temperature of 2000 F.
>
> (*e*) When increased mechanical properties are desirable, grades 8, 8c, 8t or 8F nuts may be machined from cold drawn bars without being subsequently annealed by quenching in water.

Section 4(a).—Revise to read "All nuts, excepting grades 0, 8, 8c, 8t and 8F, made by the cold process shall be . . ."

Section 5.—Add reference to new grades 8, 8c, 8t and 8F.

Table I.—Replace grade 8 with grades 8, 8c and 8t whose chemical requirements shall be the same as those for grades B8, B8c and B8t in the accompanying Table IV.

Section 14(b).—Add requirements for marking as follows:

	Marking	
Grade	Nuts Hot Forged or Cold Punched	Nuts Machined from Bar Stock
8	8	8B
8c	8c	8cB
8t	8t	8tB
8F	8F	8FB

REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends that the following standard be revised as indicated and accordingly requests the necessary nine-tenths affirmative vote at the Annual Meeting in order that these recommendations may be referred to letter ballot of the Society.

Standard Specifications for Electric-Fusion-Welded Steel Pipe for High-Temperature and High-Pressure Service (A 155 – 42):

Table I.—Revise to read as shown in the accompanying Table VII.

TABLE VII.—CHEMICAL REQUIREMENTS.

	Grade A		Grade B		Grade C	
	Ladle Analysis	Check Analysis	Ladle Analysis	Check Analysis	Ladle Analysis	Check Analysis
Carbon, max, per cent:						
for plates ¾ in. and under in thickness	0.12	0.15	0.16	0.20	0.21	0.25
for plates over ¾ in. in thickness	0.14	0.17	0.18	0.22	0.26	0.30
Manganese, per cent	0.30 to 0.60	0.27 to 0.63	0.30 to 0.60	0.27 to 0.63	0.30 to 0.60	0.27 to 0.63
Phosphorus, max, per cent	0.040	0.050	0.040	0.050	0.040	0.050
Sulfur, max, per cent	0.050	0.060	0.050	0.060	0.050	0.060
Silicon, min, per cent	0.12	0.10	0.12	0.10	0.12	0.10

WITHDRAWAL OF STANDARD

Standard Specifications for Heat-Treated Steel Elliptical Springs (A 147 – 39):

It is recommended that these specifications be discontinued because of the proposal to advance to standard the Tentative Specifications for Heat-Treated Steel Leaf Springs (A 147 - 49 T) which are intended as a replacement.

REVISION OF STANDARDS AND REVERSION TO TENTATIVES

The committee recommends revisions in the following seventeen standards with reversion to tentative.

Standard Specifications for Quenched Carbon-Steel Joint Bars (A 49 – 48):

Section 5.—In the table of chemical requirements change the maximum manganese requirement from the present "0.80" to read "1.00" per cent.

Section 11(a).—Revise to read "One tension test and one bend test shall be made from each lot of 1,000 bars or fraction thereof, but not less than one test for each heat on each day in which bars are heated and quenched."

Standard Specifications for Welded and Seamless Steel Pipe (A 53 – 47):

Section 2(a).—Under this section revise the note defining deoxidized bessemer steel deleting the words "in the ladle" after the words "and further treated." Also add a second paragraph to the note to read "Killed bessemer steel may also be deoxidized by treatment in the ladle with sufficient aluminum or its equivalent to insure practically no evolution of gases during solidification."

Section 3.—Revise to read "The steel shall conform to the requirements as to chemical composition prescribed in Table I."

New Table.—Add a Table I to read as shown in the accompanying Table VIII, renumbering subsequent tables accordingly.

Section 4.—Retain the first two sentences and revise the remainder to read "The chemical composition thus determined shall conform to the requirements prescribed in Table I."

Section 10(a).—Add a new footnote referenced after the word "length" in the third line to read "Length is defined as the length as ordered, except that in the case of orders for cut lengths shorter than single random, the term lot shall apply to the lengths as rolled, prior to cutting to the required short lengths."

TABLE VIII.—CHEMICAL REQUIREMENTS.

	Carbon, max, per cent		Phosphorus, max, per cent	
	Ladle Analysis	Check Analysis	Ladle Analysis	Check Analysis
Seamless Pipe, open-hearth	...	...	0.040	0.048
Seamless Pipe, bessemer	...	...	0.10	0.11
Furnace welded pipe, open-hearth	...	...	0.07	0.08
Furnace welded pipe, bessemer	...	...	0.12	0.13
Electric-resistance-welded open-hearth	0.10[a]	0.13[a]	0.040	0.050

[a] Applies only to sizes ⅛ and ¼ in. in diameter.

Standard Specifications for Electric-Resistance-Welded Steel Pipe (A 135 – 46):

Section 3.—Revise the chemical requirements to read as follows:

	Ladle Analysis	Check Analysis
Phosphorus, max, per cent	0.040	0.050
Sulfur, max, per cent	0.050	0.060

Section 10(a).—Revise as indicated above for Section 10(a) in Specifications A 53.

Standard Specifications for Electric-Fusion-Welded Steel Pipe (Sizes 4 in. to but not Including 30 in.) (A 139 – 46):

Section 3.—Revise as indicated above for Section 3 in Specifications A 135.

Standard Specifications for Seamless Low-Carbon and Carbon-Molyb-

denum Steel Still Tubes for Refinery Service (A 161 - 46):

Section 4(a).—Revise to read "The steel shall conform to the requirements as to chemical composition prescribed in Table I."

New Table.—Add a new Table I to read as shown in the accompanying Table IX.

Standard Specifications for Seamless Steel Boiler Tubes for High-Pressure Service (A 192 - 44):

Section 4.—Revise the table of chemical requirements as indicated above for Section 4 in Specifications A 179, and add a ladle and check analysis requirement for silicon of "0.23" and "0.25" per cent, max, respectively.

TABLE IX.—CHEMICAL REQUIREMENTS.

	Low-Carbon Steel Tubes		Carbon-Molybdenum Steel Tubes	
	Ladle Analysis	Check Analysis	Ladle Analysis	Check Analysis
Carbon, per cent	0.12 to 0.18	0.10 to 0.20	0.11 to 0.19	0.10 to 0.20
Manganese, per cent	0.33 to 0.77	0.30 to 0.80	0.33 to 0.77	0.30 to 0.80
Phosphorus, max, per cent	0.040	0.048	0.040	0.045
Sulfur, max, per cent	0.050	0.058	0.040	0.045
Silicon, per cent	0.23 max	0.25 max	0.15 to 0.45	0.10 to 0.50
Molybdenum, per cent	...	...	0.48 to 0.63	0.45 to 0.66

Standard Specifications for Electric-Resistance-Welded Steel and Open-Hearth Iron Boiler Tubes (A 178 - 47):

Table I.—Revise the chemical requirements for types A and C to read as shown in the accompanying Table X.

Standard Specifications for Seamless Cold-Drawn Intermediate Alloy-Steel Heat Exchanger and Condenser Tubes (A 199 - 46):

Table I.—Revise the chemical requirements for grades 1, 2, 3, 4, 5 and 6 to read as shown in the accompanying Table XI.

TABLE X.—CHEMICAL REQUIREMENTS.

	Type A Low-Carbon Steel		Type C Medium-Carbon Steel	
	Ladle Analysis	Check Analysis	Ladle Analysis	Check Analysis
Carbon, per cent	0.08 to 0.15	0.06 to 0.18	0.31 max	0.35 max
Manganese, per cent	0.30 to 0.60	0.27 to 0.63	0.76 max	0.80 max
Phosphorus, max, per cent	0.040	0.050	0.040	0.050
Sulfur, max, per cent	0.050	0.060	0.050	0.060

Standard Specifications for Seamless Cold-Drawn Low-Carbon Steel Heat-Exchanger and Condenser Tubes (A 179 - 46):

Section 4.—Revise the table of chemical requirements to read as follows:

	Ladle Analysis	Check Analysis
Carbon, per cent	0.08 to 0.16	0.06 to 0.18
Manganese, per cent	0.30 to 0.60	0.27 to 0.63
Phosphorus, max, per cent.	0.040	0.048
Sulfur, max, per cent	0.050	0.058

Standard Specifications for Seamless Intermediate Alloy-Steel Still Tubes for Refinery Service (A 200 - 46):

Table I.—Revise the chemical requirements for grades 1, 2, 3, 4, 5, and 6 to read as shown in the accompanying Table XI.

Standard Specifications for Seamless Carbon-Molybdenum Alloy-Steel Boiler and Superheater Tubes (A 209 – 46):

Table I.—Revise to read as shown in the accompanying Table XII.

Standard Specifications for Medium-Carbon Seamless Steel Boiler and Superheater Tubes (A 210 – 46):

Section 4(a).—Revise the table of chemical requirements to read as follows:

	Ladle Analysis	Check Analysis
Carbon, max, per cent	0.32	0.35
Manganese, max, per cent	0.77	0.80
Phosphorus, max, per cent	0.040	0.048
Sulfur, max, per cent	0.050	0.058
Silicon, min, per cent	0.12	0.10

Standard Specifications for Electric-Resistance-Welded Steel Heat-Exchanger and Condenser Tubes (A 214 – 47):

Section 4.—Revise the table of chemical requirements to read as follows:

	Ladle Analysis	Check Analysis
Carbon, max, per cent	0.15	0.18
Manganese, per cent	.30 to 0.60	0.27 to 0.63
Phosphorus, max, per cent	0.040	0.050
Sulfur, max, per cent	0.050	0.060

Standard Specifications for Electric-Resistance-Welded Steel Boiler and Superheater Tubes for High Pressure Service (A 226 – 47):

Section 4.—Revise the table of chemical requirements to read as follows:

TABLE XI.—CHEMICAL REQUIREMENTS.

	Grade 1		Grade 2		Grade 3	
	Ladle Analysis	Check Analysis	Ladle Analysis	Check Analysis	Ladle Analysis	Check Analysis
Carbon, max, per cent	0.14	0.15	0.14	0.15	0.14	0.15
Manganese, per cent	0.33 to 0.57	0.30 to 0.60	0.33 to 0.57	0.30 to 0.60	0.33 to 0.57	0.30 to 0.60
Phosphorus, max, per cent	0.025	0.030	0.025	0.030	0.025	0.030
Sulfur, max, per cent	0.025	0.030	0.025	0.030	0.025	0.030
Silicon, per cent	0.55 to 0.95	0.50 to 1.00	0.55 to 0.95	0.50 to 1.00	0.45 to 0.75	0.40 to 0.80
Chromium, per cent	1.05 to 1.45	1.00 to 1.50	2.25 to 2.75	2.15 to 2.85	1.50 to 2.00	1.45 to 2.05
Molybdenum, per cent	0.48 to 0.63	0.45 to 0.66	0.48 to 0.63	0.45 to 0.66	0.63 to 0.78	0.60 to 0.81

	Grade 4		Grade 5		Grade 6	
	Ladle Analysis	Check Analysis	Ladle Analysis	Check Analysis	Ladle Analysis	Check Analysis
Carbon, max, per cent	0.14	0.15	0.14	0.15	0.14	0.15
Manganese, per cent	0.33 to 0.57	0.30 to 0.60	0.33 to 0.57	0.30 to 0.60	0.33 to 0.57	0.30 to 0.60
Phosphorus, max, per cent	0.025	0.030	0.025	0.030	0.025	0.030
Sulfur, max, per cent	0.025	0.030	0.025	0.030	0.025	0.030
Silicon, per cent	0.45 max	0.50 max	0.45 max	0.50 max	0.45 max	0.50 max
Chromium, per cent	1.75 to 2.25	1.65 to 2.35	2.75 to 3.25	2.65 to 3.35	2.00 to 2.50	1.90 to 2.60
Molybdenum, per cent	0.48 to 0.63	0.45 to 0.66	0.83 to 1.03	0.80 to 1.06	0.90 to 1.10	0.87 to 1.13

TABLE XII.—CHEMICAL REQUIREMENTS.

	Grade T1		Grade T1a		Grade T1b	
	Ladle Analysis	Check Analysis	Ladle Analysis	Check Analysis	Ladle Analysis	Check Analysis
Carbon, per cent	0.11 to 0.19	0.10 to 0.20	0.16 to 0.24	0.15 to 0.25	0.13 max	0.14 max
Manganese, per cent	0.33 to 0.77	0.30 to 0.80	0.33 to 0.77	0.30 to 0.80	0.33 to 0.77	0.30 to 0.80
Phosphorus, max, per cent	0.040	0.045	0.040	0.045	0.040	0.045
Sulfur, max, per cent	0.040	0.045	0.040	0.045	0.040	0.045
Silicon, per cent	0.15 to 0.45	0.10 to 0.50	0.15 to 0.45	0.10 to 0.50	0.15 to 0.45	0.10 to 0.50
Molybdenum, per cent	0.48 to 0.63	0.45 to 0.66	0.48 to 0.63	0.45 to 0.66	0.48 to 0.63	0.45 to 0.66

	Ladle Analysis	Check Analysis
Carbon, per cent	0.08 to 0.15	0.06 to 0.18
Manganese, per cent	0.30 to 0.60	0.27 to 0.63
Phosphorus, max, per cent	0.040	0.050
Sulfur, max, per cent	0.050	0.060
Silicon, max, per cent	0.22	0.25

Standard Specifications for Factory-Made Wrought Carbon and Alloy Steel Welding Fittings (A 234 – 50):

Title.—Revise to read "Tentative Specifications for Factory - Made Wrought Carbon Steel and Ferritic Alloy Steel Welding Fittings."

Section 1(a).—Revise the first sentence to read "These specifications cover factory-made wrought carbon steel and ferritic alloy steel welding fittings for pressure piping."

Table I.—Revise to read as shown in the accompanying Table XIII.

TABLE XIII.—PERMISSIBLE RAW MATERIALS AND MARKING OF BUTT-WELDING FITTINGS.

Steel Composition	Grade[a]	Permissible Raw Materials[b]		
		Pipe	Plate	Forgings
Carbon steel, grade A	WPA	A 106, grade A	A 285, grade B A 285, grade C	A 105, grade I
Carbon steel, grade B	WPB	A 106, grade B	A 212, grade A	A 105, grade II
Carbon-molybdenum steel	WP1	A 206, grade P1	A 204, grade B	A 182, grade F1
$1\frac{1}{4}$ per cent chromium molybdenum steel	WP11	A 158, grade P11	[c]	[c]
$2\frac{1}{4}$ per cent chromium molybdenum steel	WP22	[d]	[d]	A 182, grade F22
5 per cent chromium molybdenum steel	WP5a	A 158, grade P5a	[e]	A 182, grade F5

[a] When fittings are of welded construction, the symbols shown above shall be supplemented by the letter "W."

[b] These designations refer to the following specifications of the American Society for Testing Materials:
Forged or Rolled Steel Pipe Flanges, Forged Fittings and Valves and Parts for High-Temperature Service (A 105 – 46),
Seamless Carbon-Steel Pipe for High-Temperature Service (A 106 – 51 T),
Seamless Alloy-Steel Pipe for High-Temperature Service (A 158 – 51 T),
Forged or Rolled Alloy-Steel Pipe Flanges, Forged Fittings and Valves and Parts for High-Temperature Service (A 182 – 49 T),
Molybdenum Steel Plates for Boilers and Other Pressure Vessels (A 204 – 50 T),
Seamless Carbon-Molybdenum Alloy-Steel Pipe for High-Temperature Service (A 206 - 51 T),
High Tensile Strength Carbon-Silicon Plates for Boilers and Other Pressure Vessels (A 212 – 50 T), and
Low and Intermediate Tensile Strength Carbon-Silicon Plates of Flange and Firebox Qualities (A285 – 50 T).

[c] No ASTM specification has as yet been written for plate or forgings of this chemical composition. However, fittings may be made from plate or forgings which conform in their mechanical and chemical properties to the pipe specification shown for this grade.

[d] No ASTM specification has as yet been written for pipe or plate of this chemical composition. However, fittings may be made from pipe or plate which conform in their mechanical or chemical properties to the forging specification shown for this grade, or to ASTM Specifications A 213, Grade T22.

[e] No ASTM specification has as yet been written for plate of this chemical composition. However, fittings may be made from plate which conforms in its mechanical and chemical properties to the pipe or forging specifications shown for this grade.

Section 6.—Insert a new Paragraph (*d*) to read as follows, relettering subsequent paragraphs accordingly:

(*d*) *Chromium-Molybdenum Steel Fittings.*—Unless otherwise specified by the purchaser and so stated in the order, chromium-molybdenum steel fittings shall be full annealed, or normalized and tempered, as described in Paragraphs (*f*) and (*i*).

In the present Paragraphs (*f*) and (*g*) delete the word "still" before the word "air."

Insert a new Paragraph (*i*) to read as follows:

(*i*) *Normalizing-Tempering.*—The procedure for normalizing and tempering shall consist in allowing the fittings, immediately after forming, to cool to a temperature below the critical range, under suitable conditions to prevent injuries by too rapid cooling. They shall then be heated to a point above the upper critical, held for the proper time to refine the grain, removed from the furnace and cooled in air. They shall then be reheated to a temperature of 1250 to 1350 F, held at temperature for not less than 1 hr per inch of thickness, and cooled slowly in the furnace or air.

Section 11(a).—Revise the second sentence to read "Each fitting shall be marked with the grade symbol for the appropriate steel composition listed in Table I."

Standard Specifications for Electric-Resistance-Welded Carbon-Molybdenum Alloy-Steel Boiler and Superheater Tubes (A 250 – 47):

Table I.—Revise to read as shown in the accompanying Table XII.

Standard Specifications for Copper Brazed Steel Tubing (A 254 – 46):

Section 4.—Revise the table of chemical requirements to read as follows:

	Ladle Analysis	Check Analysis
Carbon, per cent	0.07 to 0.12	0.05 to 0.15
Manganese, per cent	0.30 to 0.60	0.27 to 0.63
Phosphorus, max, per cent	0.040	0.050
Sulfur, max, per cent	0.050	0.060

REPORT OF COMMITTEE A-3

ON

CAST IRON*

Two developments of greatest interest in Committee A-3 during the past year have been the revision of Specification A 278 covering Gray Iron Castings for Pressure-Containing Parts for Temperatures up to 650 F to bring it in accord with ASME Boiler Code Requirements and the development of a Proposed Specification for Nodular Iron. It is expected that the latter will be proposed to the Society for publication during 1951.

Personnel.—The report of the Special Subcommittee on reclassification of personnel has been approved by Committee A-3 membership and was reflected in the classification given in the 1950 Year Book. This showed a total of 128 voting members, of whom 38 are classified as producers, 54 as consumers, and 36 as general interest members.

The committee notes with regret the retirement of W. H. Rother, Buflovak Div., Blaw-Knox Co., from active service in Committee A-3. Mr. Rother has contributed meritorious service to Committee A-3 since 1923. He was secretary from 1930 to 1933, vice-chairman from 1934 to 1935, and chairman from 1936 to 1939.

New Subcommittee.—A Subcommittee XXV on Methods of Chill Testing of Cast Iron ,was formed under the chairmanship of D. E. Krause. The membership is being organized.

REVISION OF TENTATIVE

The committee recommends that the Tentative Specifications for Gray Iron Castings for Pressure-Containing Parts for Temperatures up to 650 F (A 278 – 49 T)[1] be revised, as appended to this report.[2]

This recommendation has been submitted to letter ballot of the committee, and resulted in 78 affirmative, 0 negative, and 25 not voting ballots.

SUBCOMMITTEE ACTIVITIES

Since it is beyond the scope of this report to cover all the varied activities of Committee A-3 and its subcommittees, the following will comprise only a very brief summary.

The subcommittees responsible for tentative specifications existing for two years without revision have studied these and recommend that they remain in their tentative status.

Under consideration in the subcommittees is a proposed recommended practice for brinell hardness testing of cast iron and proposed revisions in the Automotive Gray Iron Specification A 159; also test bar specifications for chilled and gray portions of chilled iron castings.

A Research Committee of active past chairmen of Committee A-3 has reported that the project upon the study of Cast Iron for Elevated Temperature Service has been approved by the Joint Committee (A.S.M.E.–ASTM) on Effect of Temperature on the Properties of Metals, and application for financial support has been approved by the Finance Committee of that body. The project on cast iron has been assigned to the jurisdiction of the Steam Power Panel of the Joint Committee. A brochure describing the general aims of the research project is in the hands of the Joint Committee and will be used in the forthcoming campaign to raise funds.

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* Presented at the Fifty-fourth Annual Meeting of the Society, June 18–22, 1951.

[1] 1949 Book of ASTM Standards, Part 1.

[2] The revised specifications were accepted by the Society and appear in the 1951 Supplement to Book of ASTM Standards, Part 1.

This report has been submitted to letter ballot of the committee, which consists of 128 members; 85 members returned their ballots, all of whom have voted affirmatively.

Respectively submitted on behalf of the committee,

J. S. VANICK,
Chairman.

H. W. STUART,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee A-3 presented to the Society through the Administrative Committee on Standards the recommendation that Specifications for Nodular Iron Castings be published as tentative. This recommendation was accepted by the Standards Committee on November 27, 1951, and the specifications appear in the 1951 Supplement to Book of ASTM Standards, Part 1, bearing the designation A 339 – 51 T.

REPORT OF COMMITTEE A-5

ON

CORROSION OF IRON AND STEEL*

Committee A-5 on Corrosion of Iron and Steel held a meeting on June 28, 1950, during the Annual Meeting of the Society. This was the only meeting of the main committee during the year, although some of the subcommittees met on March 7, 1951, during the Spring Meeting of the Society. The committee now consists of 118 members, of whom 54 are classified as producers, 36 as consumers and 28 as general interest members.

NEW TENTATIVE

Committee A-5 recommends for publication as tentative the proposed Tentative Specifications for Zinc-Coated (Galvanized) Iron or Steel Chain Link Fence Fabric (Galvanized Before Weaving), as appended hereto.[1]

REVISION OF TENTATIVE

Committee A-5 joins with Committee B-8 in recommending revisions in Tentative Methods of Test for Local Thickness of Electrodeposited Coatings (A 219 - 45 T)[2], which is under their joint jurisdiction. Revise Sections A1, A2, A3, and A4 of Appendix I as follows:

Section A1.—Delete the present Paragraph (*a*) and add the following:

> (*a*) *Nature of Test.*—The magnetic methods for estimation of thickness of coatings on metals involve instruments which measure either the attractive force between a magnet and the coated specimen or the reluctance of a flux path through the coating and the basis metal. Either the attractive force or the reluctance as measured, is a function of the thickness of the coatings. These methods may be used to measure the thickness of nonmagnetic or nickel coatings on a magnetic basis metal such as steel, the thickness of magnetic coatings on a nonmagnetic basis metal, and the thickness of the nickel and copper in a composite nickel over copper coating on steel.

In Paragraph (*b*) change "principles" to "types of apparatus." Delete the second sentence, and delete the footnotes for all three items in Paragraph (*b*). To Item (*1*) add "At this time this type is the only one suitable for measuring nickel coatings and composite nickel-over-copper coatings on steel." In Item (*3*) change "electromagnetic" to "magnetic."

Delete the present Paragraph (*c*).

Section A2.—Revise the introduction on Factors Affecting Applicability and Accuracy of Measurement to read as follows by the addition of the italicized words: "The following conditions *may* affect the result obtained *by a given instrument.*"

In Items (*1*), (*2*), and (*3*), change "base" to "basis". In Item (*3*) add the words "and residual magnetism." In Item (*6*) after the word "shape" add the words "and strength."

Add new Item (*10*) to read as follows: "Grain orientation in the steel basis metal."

Section A3.—Revise to read as follows:

> (*a*) *Calibration.*—Each instrument, including the magnet to be used, shall be calibrated before use against suitable standards and the calibration should be checked at frequent intervals.
>
> (*b*) Thickness standards for electrodeposited coatings on flat smooth surfaces may be purchased from the National Bureau of Standards.

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* Presented at the Fifty-fourth Annual Meeting of the Society, June 18-22, 1951.

[1] The new tentative was accepted by the Society and appears in the 1951 Supplement to Book of ASTM Standards, Part 1.

[2] 1949 Book of ASTM Standards, Part 1.

(*c*) When surfaces or coatings to be tested deviate from the standards described in Section A3 (*b*), as indicated in A2, the standards used shall be such that errors due to the factors listed in Section A2 will be small enough to permit the accuracy requirements of Section A4 to be met. The thickness of such standards shall be determined by the microscopic method, or by stripping methods when applicable.

Section A4.—Revise to read as follows:

Accuracy.—The instrument, its calibration, and its operation shall be such that the coating thickness can be determined within ±5 per cent of its true thickness for single coatings and ±15 per cent for composite coatings.

Delete the note in Section A4.

ADOPTION OF TENTATIVE AS STANDARD

The committee recommends that the Specifications for Zinc-Coated (Galvanized) "Iron" Telephone and Telegraph Line Wire (A 111 - 49 T)[2] be approved for reference to letter ballot of the Society for adoption as standard without revision.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation as tentative of the following specifications without revision:

Tentative Specifications for:

Lead-Alloy Coating (Hot-Dip) on Iron or Steel Hardware (A 267 - 46 T).—In the 1950 report of Committee A-5[3] it was recommended that these specifications remain tentative because there was little use made of lead-coated hardware and further decline in its use was anticipated which would warrant dropping the specifications altogether in a few years. However, the anticipated decline has not materialized, doubtless because of the present shortage of metals. It appears, therefore, that more use will be made of A 267 in the immediate future than has been the case in the past few years.

Zinc-Coated (Galvanized) High Tensile Steel Telephone and Telegraph Line Wire (A 326 - 49 T).—Subcommittee XII, which is responsible for these specifications, is considering the desirability of adding a third grade, 190, to these specifications; therefore, it is considered best to retain them as tentative, pending a decision on the matter.

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee VII on Methods of Test (W. E. Buck, chairman) is considering making minor changes in several methods of test to permit square, rectangular, or circular test specimens and to remove from these methods of test specific instructions concerning the locations from which the test specimens are to be taken. The study on the reactions of various coatings (hot-dipped, electrodeposited, heat-treated, etc.) in copper-sulfate solutions is continuing.

Subcommittee XI on Sheet Specifications (E. F. Lundeen, chairman) has prepared a draft of a new tentative specification for 1.25-oz. zinc-coated (galvanized) iron or steel roofing sheets which will be sent out shortly for letter ballot in the subcommittee. Proposed changes in the Tentative Specifications for Zinc-Coated (Galvanized) Iron or Steel Sheets, Coils and Cut Lengths (A 93 - 50 T),[5] involving the Bend Test on heavy gages, the Sheet Weight Test, and the Ordered Weight of Coating are also being considered.

Subcommittee XII on Wire Specifications (P. E. Chisler, chairman) prepared the new Tentative Specifications for Zinc-Coated (Galvanized) Iron or Steel Chain-Link Fence Fabric (Galvanized Before Weaving) which are being recommended to the Society for publication. The subcommittee is considering the inclusion of a tolerance limit on the wire in the fabric of the Standard Specifications for Zinc-Coated Iron or Steel Chain-Link Fence Fabric Galvanized After Weaving (A 117 - 33),[2] as has been done for the proposed tentative for fabric galvanized before weaving. Consideration is being given to the addition of the 190,000-psi grade to Tentative Specifications for Zinc-Coated (Galvanized) High Tensile Strength Steel Telephone and Telegraph Line Wire (A 326 - 49 T).[2]

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[3] *Proceedings*, Am. Soc. Testing Mats., Vol. 50, p. 100 (1950).

[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

[5] 1950 Supplement to 1949 Book of ASTM Standards, Part 1

Subcommittee XIII on Hardware Specifications (B. J. Barmack, chairman) is conducting a letter ballot at the present time on revisions of Standard Specifications for Zinc (Hot-Galvanized) Coating on Structural Steel Shapes, Plates and Bars, and Their Products (A 123 - 47)[2] and Standard Specifications for Zinc-Coating (Hot-Dip) on Iron and Steel Hardware (A 153 - 49).[2] These revisions will clarify the use of these two specifications insofar as small structural shapes, commonly called bar sizes, bolts, and washers, are concerned and will eliminate the uncertainty which exists at present as to which one of these specifications is the proper one for these "borderline" items. Further study is being made toward the formulation of a simple embrittlement or malleability test applicable to hardware. It is also intended to add an Appendix to Specifications A 153 on the use of low-alloy steel for pole line hardware, explaining that it will considerably increase the service life of this material and will in addition increase the weights of zinc coatings above the minimum values shown in Table I of these specifications.

Subcommittee XIV on Sheet Tests (E. S. Taylerson, chairman) has made the annual inspection of the black sheets at Annapolis and the galvanized sheets at Altoona, Pittsburgh, Sandy Hook, State College, and Key West. The biannual report will be published in 1952.

Subcommittee XV on Wire Tests (A. P. Jahn, chairman) makes its report as appended hereto under the title, "Report of Subcommittee XV on Field Tests of Wire and Wire Products."

Subcommittee XVI on Hardware Tests (A. Mendizza, chairman) is continuing its yearly inspections of hardware undergoing exposure tests and, in accordance with their reporting schedule, will make a formal report next year.

This report has been submitted to letter ballot of the committee, which consists of 118 members; 62 members returned their ballots, of whom 60 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

T. R. GALLOWAY,
Chairman.

H. F. HORMANN,
Secretary.

REPORT OF SUBCOMMITTEE XV ON FIELD TESTS OF WIRE AND WIRE PRODUCTS

(Covers 1950 Inspections)

This report summarizes the results of the atmospheric corrosion tests on wire and wire products through 1950. Reference to earlier reports of Committee A-5 on Corrosion of Iron and Steel, which appeared in the Society's *Proceedings* for 1939, 1941, 1943, 1945, 1947, and 1949, will be helpful in studies of these exposure test results. These earlier reports contain descriptions of the test methods and micrographs and other characterizations of specimens in the test as well as the data assembled during previous inspections.

The materials under test are mostly bare steel and zinc-coated wires and wire products but copper-covered and lead-coated steel wires and chromium and chromium-nickel steel wires are also included. The coatings are expressed in weight terms of ounces per square foot of surface. For ready comparisons of coating thickness, 1 oz per sq ft of surface may be considered as averaging 0.0017 in. thick for zinc coatings, 0.0010 in. thick for lead[1] coatings and 0.0013 in. thick for copper coatings.

Specimens of wires and wire products have been exposed for about 14 years at eleven locations. The location of these sites and their general classification regarding atmospheres are as follows:

Test Location	General Type of Atmosphere	Remarks
Pittsburgh, Pa.	Severe industrial	On Brunot Island in the Ohio River about 2 miles west of the city.
Bridgeport, Conn.	Industrial	In the city.
Sandy Hook, N. J.	Seacoast	About 300 yd. from Atlantic Ocean.
Santa Cruz, Calif.	Seacoast	About 3 miles from Pacific Ocean.
State College, Pa.	Rural	Central Pennsylvania.
Lafayette, Ind.	Rural	Wabash River Valley.
Ames, Iowa	Rural	Central Iowa.
Manhattan, Kans.	Rural	Kansas River Valley.
Ithaca, N. Y.	Rural	Central New York.
College Station, Tex.	Rural	About 120 miles inland.
Davis, Calif.	Rural	About 80 miles inland.

At each location, more than 900 specimens were exposed. These included short lengths of wire (42 in. long) and wire strand at all locations; farm field fence at nine sites; barbed wire at eight locations; and chain link fence at eight locations.

The extent of corrosion is being measured by visual examination of all specimens at the test site and by tension tests on the unfabricated wire specimens. During 1949 and 1950 the Pittsburgh, Bridgeport, Sandy Hook, and State College specimens were inspected in the spring of each year by a traveling committee. Inspections were made in the fall of 1949 and fall of 1950 at the other test sites by the university people in charge of the test plots.

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[1] The lead-coated wires in these tests were lead coated over a bonding coat of zinc. See *Proceedings*, Am. Soc. Testing Mats., Vol. 43, p. 87 (1943), for analysis of lead coatings.

TABLE A.—CORROSION CONDITIONS OF ZINC-COATED UNFABRICATED WIRES AFTER APPROXIMATELY 14 YEARS' EXPOSURE.

R = rust; Y = yellowed; G = gray; M = metallic; MG and GY = intermediate states; D = dark to exclusion of better observation; SR and SY = superficial R and Y; R+Y = indistinguishable proportions of R and Y; Gn = green; Br = brown; PHR and PHY = rust or yellowed in pin holes.

Location	Coating Group, oz per sq ft of surface										
	0.20 to 0.30	0.25 to 0.35	0.35 to 0.45	0.45 to 0.55	0.50 to 0.60	0.60 to 0.70	0.70 to 0.85	0.80 to 1.00	1.20 to 1.35	1.60 to 1.85	2.00 or more
Pittsburgh, Pa.	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R
Sandy Hook, N. J.	100R	100R	100R	100R	100R	100R	100R	100R	98R	12R	G
Bridgeport, Conn.	100R	100R	100R	100R	100R	100R	100R	100R	99R	25R	G
State College, Pa.	100R	100R	100R	100R	97R	80R	65R	25R	GY	G	G
Lafayette, Ind.	100R	100R	100R	100R	100R	99R	99R	80R	GY	G	G
Ames, Iowa	100R	100R	100R	45R	60R	6R	5R	1R	G	G	G
Manhattan, Kans.	55R	45R	1R	GY	G	G	MG	MG	MG	MG	MG
Ithaca, N. Y.	100R	100R	100R	100R	100R	98R	95R	45R	GY	G	MG
Santa Cruz, Calif.	40R	25R	10R	5R	3R	5R	2R	1R	...	MG	MG
College Sta., Tex.	95R	60R	5R	15R	3R	10R	3R	2R	G	G	G
Davis, Calif.	10R	10R	1R	G	G	G	G	G	...	MG	MG

TABLE B.—CORROSION CONDITIONS OF UNFABRICATED WIRES CARRYING COATINGS OTHER THAN ZINC—AFTER APPROXIMATELY 14 YEARS' EXPOSURE.

R = rust; Y = yellowed; G = gray; M = metallic; MG and GY = intermediate states; D = dark to exclusion of better observation; SR and SY = superficial R and Y; R+Y = indistinguishable proportions of R and Y; Gn = green; Br = brown; PHR and PHY = rust or yellowed in pin holes.

Location	Coating Groups and Corrosion-Resistant Groups				
	Copper-Covered Wires[a]	Lead-Coated Wires[b]	12–14% Chromium Cold Drawn	12–14% Chromium Air Quenched	18% Chromium 8% Nickel
Pittsburgh, Pa.	D	D	D	D	D
Sandy Hook, N. J.	Gn	100R+Y	SR	YSR	MG
Bridgeport, Conn.	Gn	GY	Y	GY	MG
State College, Pa.	Gn	100R+Y	MG	MG	M
Lafayette, Ind.	Gn	50PHR	M	M	M
Ames, Iowa	Br	25R	SY	SY	SY
Manhattan, Kans.	Br	5R	MG	MG	M
Ithaca, N. Y.	Br	10R	SY	SY	M
Santa Cruz, Calif.	Br	30SR	80SR	35SR	MG
College Sta., Tex.	Br	30R+Y	99R+Y	85R+Y	MG
Davis, Calif.	Br	35SR	50SR	25SR	MG

[a] Weight of coating, 7 to 9 oz per sq ft of surface.
[b] Weight of coating, 1.4 to 1.9 oz per sq ft of surface.

TABLE C.—CORROSION CONDITIONS OF FARM FIELD FENCE AFTER APPROXIMATELY 14 YEARS' EXPOSURE.

R = rust; Y = yellowed; R+Y = indistinguishable proportions of R and Y; G = gray; M = metallic; MG and GY = intermediate state; PHR, PHY = pin hole rusting or yellowing; SR, SY = superficial rust or yellowing; Gn = green; Br = brown.

Location	Coating Group, oz zinc per sq ft of surface							Copper-Covered[a]	Lead-Coated[b]	Chromium-Nickel Steel
	0.20 to 0.30	0.25 to 0.35	0.35 to 0.45	0.45 to 0.55	0.50 to 0.60	0.60 to 0.70	0.70 or more			
State College, Pa.	100R	100R	100R	100R	80R	65R	10R	Gn	100R+Y	MG
Lafayette, Ind.	100R	100R	100R	100R	95R	80R	15R	Gn	15PHR	M
Ames, Iowa	100R	100R	70R	25R	5R	G	G	Br	25R	SY
Manhattan, Kans.	40R	25R	5R	GY	G	G	MG	Br	20R	MG
Ithaca, N. Y.	100R	100R	90R	80R	60R	50R	15R	Gn	10R	MG
Santa Cruz, Calif.	35R	30R	20R	20R	5R	5R	1R	Br	40SR	50SR
College Sta., Tex.	65R	40R	30R	10R	10R	5R	1R	Br	15R	10SR
Davis, Calif.	20R	30R	G	G	G	G	MG	Br	40R	40SR

[a] Weight of coating, 7.42 oz per sq ft of surface.
[b] Weight of coating, 1.48 oz per sq ft of surface.

TABLE D.—CORROSION CONDITIONS OF BARBED WIRE AFTER APPROXIMATELY 14 YEARS' EXPOSURE.

R = rust; Y = yellowed; R+Y = indistinguishable proportions of R and Y; G = gray; M = metallic; MG and GY = intermediate state; PHR, PHY = pin hole rusting or yellowing; SR, SY = superficial rust or yellowing; Gn = green Br = brown.

Location	Coating Group, oz zinc per sq ft of surface							Copper-Covered[a]	Lead-Coated[b]
	0.20 to 0.30	0.35 to 0.45	0.50 to 0.60	0.70 to 0.85	0.80 to 1.00	1.60 to 1.80	2.00 or more		
State College, Pa.	100R	100R	65R	G	GY	G	G	Gn	....
Lafayette, Ind.	100R	100R	100R	98R	10R	10R	15R	Gn	15PHR
Ames, Iowa	100R	80R	5R	G	G	G	G	Br	20R
Manhattan, Kans.	40R	10R	10R	MG	MG	MG	MG	Br	15PHY
Ithaca, N. Y.	100R	95R	90R	35R	1R	G	M	Gn	10R
Santa Cruz, Calif.	30R	15R	10R	G	G	G	MG	Br	30SR
College Sta., Tex.	75R	25R	10R	G	G	G	M	Br	4R
Davis, Calif.	10R	20R	10R	MG	MG	MG	MG	Br	25SR

[a] Weight of coating, 3.97 oz per sq ft of surface.
[b] Weight of coating, 1.83 oz per sq ft of surface.

TABLE E.—CORROSION CONDITIONS OF CHAIN LINK FENCE AFTER APPROXIMATELY 14 YEARS' EXPOSURE.

R = rust; Y = yellowed; R+Y = indistinguishable proportions of R and Y; G = gray; M = metallic; MG and GY = intermediate state; PHR, PHY = pin hole rusting or yellowing; SR, SY = superficial rust or yellowing; Gn = green; Br = brown; D = dark (dirt or soot) to the exclusion of a better observation.

Location	Coating Group, oz zinc per sq ft of surface					Copper-Covered[c]	Lead-Coated[d]	Corrosion-Resistant
	0.29 to 0.52[a]	1.13 to 1.22[a]	2.09 to 2.92[a]	1.40 to 1.97[b]	4.21 or more[b]			
Pittsburgh, Pa.	100R	100R	100R	100R	100R	...	100R	D
Sandy Hook, N. J.	100R	100R	G	95R	GY	Gn	50R+Y	G
Bridgeport, Conn.	100R	100R	G	85R	Y	Gn	Y	MG
State College, Pa.	98R	GY	G	GY	G	Gn	100R+Y	M
Lafayette, Ind.	100R	GY	MG	1R	GY	Gn	100R+Y	M
Ithaca, N. Y.	95R	GY	MG	GY	G	Br	100R+Y	M
Santa Cruz, Calif.	15R	MG	MG	MG	MG	Br	35SR	MG
Davis, Calif.	10R	MG	MG	MG	MG	Br	35SR	MG

[a] Coated before weaving.
[b] Coated after weaving.
[c] Weight of coating, 8.52 oz per sq ft of surface.
[d] Weight of coating, 1.75 oz per sq ft of surface.

TABLE F.—CORROSION CONDITIONS OF WIRE STRAND AFTER APPROXIMATELY 14 YEARS' EXPOSURE.

R = rust; Y = yellowed; R+Y = indistinguishable proportions of R and Y; G = gray; M = metallic; MG and GY = intermediate state; PHR, PHY = pin hole rusting or yellowing; SR, SY = superficial rust or yellowing; Gn = green; Br = brown; D = dark (dirt or soot) to the exclusion of a better observation.

Location	Coating Group, oz zinc per sq ft of surface				Lead-Coated[a]
	0.69 to 0.93	1.03 to 1.17	1.32 to 1.51	1.71 to 2.47	
Pittsburgh, Pa.	100R	100R	100R	100R	100R+Y
Sandy Hook, N. J.	50R	25R	5R	G	100R+Y
Bridgeport, Conn.	60R	15R	5R	GY	10R
State College, Pa.	G	G	G	G	100R+Y
Lafayette, Ind.	1R	GY	GY	G	50PHR
Ames, Iowa	G	G	G	G	40R
Manhattan, Kans.	MG	MG	MG	MG	10R
Ithaca, N. Y.	1R	G	GY	MG	20R
Santa Cruz, Calif.	1R	MG	MG	MG	60SR
College Sta., Tex.	G	G	G	G	15R
Davis, Calif.	G	MG	G	MG	10R

[a] Weight of coating, 1.96 oz per sq ft of surface.

TABLE I.—REPORT OF INSPECTIONS

Abbreviations and symbols used to designate appearance or states of corrosion: M = metallic, G = gray, Y = yellowed, or rust stained, but not showing actual rough rust of base metal. R = rust of base metal. PHR, PHY = rust or yellowed in pinholes. B, Br, Gn, respectively, signify corroded to a black, brown, or green appearance, D = dark (dirt or soot) to the exclusion of a better observation. Cr = yellow from chromate treatment. MG = intermediate between M and G. Gy = predominately gray but showing indication of Y. Figures (as 5, 20, 50, 100) are percentages.

Coating Group	Average Coating, oz. per sq.ft.of Surface[a]	Wire	State College, Pa.		Lafayette, Ind.		Ames, Iowa		Manhattan, Kans.		Ithaca, N.Y.		Santa Cruz, Cal.		College Sta.,Tex.		Davis, Cal.	
			12.30 Yr.	13.30 Yr.	13.00 Yr.	14.00 Yr.	12.92 Yr.	13.85 Yr.	12.92 Yr.	13.90 Yr.	12.93 Yr.	13.93 Yr.	12.83 Yr.	13.84 Yr.	12.84 Yr.	13.87 Yr.	12.84 Yr.	13.85 Yr.
0.20	0.27	No. 512	100R	100R	100R	100R	100R	100R	60R	97R	100R	100R	39R	71R	100R	100R	16R	21R
to	0.30	No. 410	100R	100R	100R	100R	100R	100R	80R	80R	100R	100R	39R	64R	100R	100R	17R	31R
0.30	0.28	No. 411	100R	100R	100R	100R	100R	100R	10R	25R	100R	100R	5R	12R	38R	71R	1R	1R
Avg.	0.29	No. 510	100R	100R	100R	100R	100R	100R	10R	35R	100R	100R	4R	15R	70R	96R	5R	5R
0.28	0.29	No. 412	100R	100R	100R	100R	100R	100R	GY	5R	100R	100R	5R	15R	77R	97R	GY	GY
	0.24	No. 511	100R	100R	100R	100R	100R	100R	90R	95R	100R	100R	8R	29R	100R	100R	6R	7R
0.25	0.25	No. 315	100R	100R	100R	100R	100R	100R	90R	90R	100R	100R	54R	71R	100R	100R	36R	40R
to	0.28	No. 215	100R	100R	100R	100R	100R	100R	80R	80R	100R	100R	38R	44R	100R	100R	19R	26R
0.35	0.27	No. 117	100R	100R	100R	100R	100R	100R	40R	80R	100R	100R	9R	15R	100R	100R	10R	12R
Avg.	0.38	No. 317	100R	100R	100R	100R	99R	100R	GY	GY	100R	100R	3R	4R	8R	13R	G	G
0.30	0.36	No. 217	100R	100R	100R	100R	100R	100R	10R	20R	100R	100R	4R	11R	19R	28R	G	G
	0.32	No. 316	100R	100R	100R	100R	100R	100R	5R	20R	100R	100R	6R	12R	11R	18R	1R	2R
	0.30	No. 216	100R	100R	100R	100R	100R	100R	GY	GY	100R	100R	7R	15R	9R	21R	GY	GY
	0.25	No. 115	100R	100R	100R	100R	100R	100R	80R	75R	100R	100R	29R	36R	95R	98R	14R	18R
0.35	0.36	No. 520	100R	100R	100R	100R	100R	100R	GY	GY	100R	100R	7R	13R	44R	83R	1R	1R
to	0.35	No. 422	100R	100R	100R	100R	100R	100R	GY	GY	100R	100R	4R	7R	6R	10R	G	G
0.45	0.31	No. 423	100R	100R	100R	100R	100R	100R	GY	GY	100R	100R	5R	9R	5R	9R	G	G
Avg.	0.44	No. 522	100R	100R	100R	100R	100R	100R	GY	GY	100R	100R	5R	7R	6R	10R	G	G
0.38	0.37	No. 282	100R	100R	100R	100R	100R	100R	GY	GY	100R	100R	2R	5R	7R	12R	G	G
	0.38	No. 521	100R	100R	100R	100R	100R	100R	GY	GY	100R	100R	8R	11R	8R	13R	G	G
	0.47	No. 421	100R	100R	100R	100R	100R	100R	5R	5R	100R	100R	7R	15R	7R	14R	G	G
0.45	0.44	No. 325	100R	100R	100R	100R	25R	72R	5GY	GY	100R	100R	5R	8R	7R	11R	G	G
to	0.50	No. 228	100R	100R	100R	100R	67R	97R	GY	GY	100R	100R	6R	9R	9R	13R	G	G
0.55	0.54	No. 329	100R	100R	100R	100R	20R	28R	2R	GY	100R	100R	7R	10R	10R	14R	G	G
Avg.	0.52	No. 227	100R	100R	100R	100R	5R	24R	GY	GY	100R	100R	5R	9R	9R	11R	G	G
0.47	0.41	No. 125	100R	100R	100R	100R	100R	100R	GY	GY	100R	100R	6R	10R	16R	22R	G	G
	0.54	No. 127	100R	100R	100R	100R	8R	24R	GY	GY	100R	100R	5R	8R	9R	15R	G	G
	0.38	No. 326	100R	100R	100R	100R	40R	59R	GY	GY	100R	100R	1R	1R	7R	12R	G	G
	0.45	No. 226	100R	100R	100R	100R	6R	16R	GY	GY	100R	100R	2R	4R	9R	11R	G	G
	0.47	No. 327	100R	100R	100R	100R	9R	39R	GY	GY	100R	100R	3R	5R	[illegible]	7R	G	G
	0.45	No. 225	100R	100R	100R	100R	1R	11R	GY	GY	100R	100R	4R	6R	6R	10R	G	G
0.50	0.61	No. 530	35R	90R	100R	100R	14R	85R	MG	MG	100R	100R	MG	MG	G	G	MG	MG
to	0.57	No. 432	50R	95R	100R	100R	1R	11R	MG	MG	100R	100R	MG	MG	G	G	MG	MG
0.60	0.60	No. 531	100R	100R	100R	100R	93R	100R	G	G	100R	100R	MG	MG	G	G	G	G
Avg.	0.48	No. 431	100R	100R	100R	100R	77R	100R	G	GY	100R	100R	5R	8R	51R	11R	G	G
0.56	0.54	No. 430	100R	100R	100R	100R	6R	7R	G	GY	100R	100R	5R	7R	21R	7R	G	G
0.60	0.69	No. 338	5R	10R	90R	100R	G	G	MG	MG	59R	100R	MG	MG	G	G	MG	MG
to	0.68	No. 236	G	5R	96R	100R	G	G	MG	MG	52R	99R	MG	MG	G	G	MG	MG
0.70	0.61	No. 335	100R	100R	100R	100R	3R	46R	G	G	100R	100R	MG	MG	G	G		G
Avg.	0.63	No. 237	60R	80R	96R	99R	1R	4R	MG	MG	69R	85R	MG	MG	G	G	MG	MG
0.65	0.74	No. 135	50R	90R	100R	100R	G	G	G	G	94R	99R	MG	MG	G	G	G	G
	0.57	No. 337	99R	100R	100R	100R	6R	5R	GY	GY	100R	100R	6R	9R	11R	15R	G	G
	0.67	No. 235	30R	80R	96R	100R	G	G	GY	GY	75R	95R	4R	4R	8R	11R	G	G
	0.65	No. 239	95R	100R	100R	100R	1R	2R	GY	GY	100R	100R	4R	7R	7R	11R	G	G
	0.67	No. 138	95R	100R	100R	100R	5R	4R	GY	GY	99R	100R	6R	9R	12R	15R	G	G
	0.64	No. 139	75R	95R	100R	100R	5R	6R	GY	GY	92R	99R	6R	10R	14R	19R	G	G
	0.66	No. 336	90R	100R	100R	100R	4R	4R	GY	GY	100R	100R	6R	9R	10R	13R	G	G
	0.60	No. 238	95R	100R	100R	100R	2R	4R	GY	GY	100R	100R	6R	8R	10R	13R	G	G
0.70	0.70	No. 540	G	15R	100R	100R	G	G	MG	MG	29R	100R	MG	MG	G	G	G	G
to	0.72	No. 442	G	15R	100R	100R	G	G	MG	MG	67R	100R	MG	MG	G	G	MG	MG
0.85	0.79	No. 543	30R	50R	85R	94R	6R	15R	MG	MG	39R	72R	G	G	G	G	G	G
Avg.	0.78	No. 441	25R	95R	100R	100R	5R	6R	MG	MG	29R	98R	G	G	G	G	G	G
0.75	0.70	No. 542	92R	100R	100R	100R	G	15R	G	G	100R	100R	MG	MG	G	G	G	G
	0.74	No. 443	35R	95R	100R	100R	2R	2R	MG	MG	77R	97R	MG	MG	G	G	G	G
	0.74	No. 495	Y	Y	98R	100R	G	G	GY	G	19R	91R	-	-	Cr	Cr	-	-
	0.76	No. 541	92R	100R	100R	100R	G	G	G	G	100R	100R	7R	8R	7R	13R	G	G
	0.76	No. 440	100R	100R	100R	100R	G	G	GY	GY	100R	100R	6R	8R	5R	10R	G	G
	0.87	No. 444	50R	90R	100R	100R	G	G	GY	GY	46R	96R	4R	5R	5R	8R	G	G
0.80	1.05	No. 348	G	G	G	G	G	G	MG	MG	MG	G	MG	MG	G	G	MG	MG
to	0.81	No. 248	G	10R	100R	100R	G	G	MG	MG	70R	100R	MG	MG	G	G	MG	MG
1.00	0.88	No. 347	Y	1R	14R	55R	1R	1R	MG	MG	6Y	1R	G	G	G	G	G	G
Avg.	0.95	No. 246	GY	1R	19R	31R	G	G	MG	MG	6Y	6R	G	G	G	G	MG	MG
0.88	0.88	No. 346	Y	35R	100R	100R	G	G	G	G	1R	71R	G	G	G	G	G	G
	0.81	No. 245	50R	85R	99R	99R	G	G	G	G	81R	98R	G	MG	G	G	G	G
	0.81	No. 296	Y	Y	14R	91R	G	G	G	G	1R	7R	-	-	-	-	-	-
	0.90	No. 148	Y	5R	92R	94R	G	G	G	MG	1R	61R	MG	MG	G	G	G	G
	0.85	No. 149	5R	20R	88R	88R	G	5R	G	MG	11R	52R	G	G	G	G	MG	MG
	0.84	No. 345	50R	90R	100R	100R	G	G	GY	GY	48R	83R	4R	4R	5R	10R	G	G
	0.96	No. 247	1R	10R	96R	100R	G	G	GY	GY	1R	9R	3R	3R	5R	10R	G	G
Avg.	1.35	No. 800	GY	GY	GY	Y	G	G	G	MG	GY	GY	-	-	G	G	-	-
1.28	1.20	No. 801	GY	GY	GY	Y	G	G	G	MG	GY	GY	-	-	G	G	-	-
1.60	2.08	No. 552	G	G	G	G	G	G	MG	MG	G	G	MG	MG	G	G	MG	MG
to	1.83	No. 451	G	G	G	G	G	G	MG	MG	G	G	MG	MG	G	G	MG	MG
1.80	1.75	No. 350	G	G	G	G	G	G	MG	MG	G	G	MG	MG	G	G	MG	MG
Avg.	1.69	No. 252	G	G	G	G	G	G	G	MG	MG	MG	MG	MG	G	G	MG	MG
1.76	1.65	No. 351	G	G	1R	GY	G	G	MG	MG	G	G	G	G	G	G	MG	MG
	1.76	No. 250	G	G	G	G	G	G	MG	MG	G	G	G	G	G	G	G	G
	1.69	No. 551	G	G	G	G	G	G	MG	MG	G	G	G	G	G	G	MG	MG
	1.50	No. 450	G	G	G	GY	G	G	MG	MG	G	G	G	MG	G	G	MG	MG
	1.75	No. 352	G	G	GY	GY	G	G	MG	MG	G	G	G	G	G	G	MG	MG
	1.86	No. 251	G	G	G	GY	G	G	MG	MG	G	G	G	G	G	G	MG	MG
	1.86	No. 151	G	G	GY	GY	G	G	G	MG	G	G	MG	MG	G	G	MG	MG
Heavy	2.80	No. 397	G	G	G	G	G	G	MG	MG	MG	MG	MG	MG	G	G	MG	MG
Avg.	2.72	No. 298	G	G	G	G	G	G	MG	MG	MG	MG	MG	MG	G	G	MG	MG
2.89	3.00	No. 299	G	G	G	G	G	G	MG	MG	MG	G	-	-	G	G	-	-

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[a] The zinc coating weight reported in this column is the average of all stripping test determinations made on samples of unfabricated wire, fence or barbed wire of the specific wire lot number.

SPECTIONS OF UNFABRICATED WIRE SPECIMENS.

Abbreviations and symbols used to designate appearance or states of corrosion: M = metallic, G = gray, Y = yellowed, or rust stained, but not showing actual rough rust of base metal. (In cases of zinc coatings, the G and Y conditions are characteristic states of corrosion of zinc iron alloys in the coatings.) R = rust of base metal. MG = intermediate between M and G. GY predominately gray but showing indication of Y. Figures (as 5, 20, 50, 100) are percentages.

Coating Group[b]	Average Coating, oz. per sq. ft. of surface[a]	Wire Lot	SANDY HOOK, N.J.		BRIDGEPORT, CONN.	
			12.44 Yr.	13.44 Yr.	12.44 Yr.	13.44 Yr.
Avg.	1.35	No. 800.....	90R	95R	98R	99R
1.28	1.20	No. 801.....	95R	100R	100R	100R
1.60	2.08	No. 552.....	G	G	G	G
to	1.83	No. 451.....	G	G	G	30R
1.80	1.75	No. 350.....	G	G	G	G
	1.69	No. 252.....	G	G	G	10R
	1.65	No. 351.....	5R	10R	10R	40R
	1.76	No. 250.....	1R	5R	10R	20R
	1.69	No. 551.....	G	G	G	10R
	1.50	No. 450.....	10R	30R	75R	99R
	1.75	No. 352.....	10R	10R	10R	20R
	1.86	No. 251.....	30R	30R	10R	10R
Avg. 1.76	1.86	No. 151.....	25R	50R	10R	30R
Heavy	2.80	No. 397.....	G	G	G	G
Avg.	2.72	No. 298.....	G	G	G	G
2.89	3.00	No. 299.....	G	G	G	G

a. The zinc coating weight reported in this column is the average of all stripping test determination made on samples of unfabricated wire, fence or barbed wire of the specific wire lot number.

b. All other coating weight groups were 100R.

Abbreviations and symbols used to designate appearance or states of corrosion: M = metallic, G = gray, Y = yellowed, R = rust of base metal, PHR, PHY = rust or yellowed in pinholes. B, Br, Gn, respectively, signify corroded to a black, brown, or green appearance. D = dark (dirt or soot) to the exclusion of a better observation. MG = intermediate between M and G. GY predominately gray but showing indication of Y. SY* or SR* indicate, respectively: superficial Y and superficial, R, the asterisk denoting a speckled appearance. Figures (as 5, 20, 50, 100) are percentages. R+Y = indistinguishable proportions of R and Y.

	Gage	Wire	Sandy Hook, N.J.		Bridgeport, Conn.		State College, Pa.		College Sta., Texas		Davis, Calif.	
			12.44 Yr.	13.44 Yr.	12.45 Yr.	13.45 Yr.	12.30 Yr.	13.30 Yr.	12.84 Yr.	13.87 Yr.	12.84 Yr.	13.85 Yr.
Copper-covered wire	11	No. 021, 7.42-oz.	Gn	Gn	Gn	Gn	Gn-B	Gn-B	Br	Br	Br	Br
	9	No. 022, 9.24-oz.	Gn	Gn	Gn	Gn	Gn-B	Gn-B	Br	Br	Br	Br
Lead-coated Wires	6	No. 040, 1.40-oz.	100R+Y	100R+Y	GY	GY	100R+Y	100R+Y	12PHY	21R+Y	22SR	22SR
	9	No. 041, 1.48-oz.	100R+Y	100R+Y	GY	GY	100R+Y	100R+Y	33PHY	56R+Y	60SR	65SR
	11	No. 042, 1.68-oz.	100R+Y	100R+Y	GY	GY	100R+Y	100R+Y	23PHY	35R+Y	24SR	24SR
	14-1/2	No. 043, 1.90-oz.	100R+Y	100R+Y	GY	GY	100R+Y	100R+Y	2PHY	6R+Y	19SR	19SR
Corrosion-Resistant Uncoated Wires:												
12-14% Cr., Cold drawn	12	No. 001	YSR	SR	GY	Y	MG	MG	100R+Y	100R+Y	49SR	49SR
	9	No. 004	YSR	SR	GY	Y	MG	MG	96R+Y	98R+Y	45SR	46SR
	17	No. 005	YSR	SR	GY	Y	MG	MG	100R+Y	100R+Y	54SR	55SR
12-14% Cr., air quenched	13	No. 002	YSR	YSR	GY	GY	MG	MG	86R+Y	88R+Y	19SR	19SR
	10	No. 006	YSR	YSR	GY	GY	MG	MG	63R+Y	78R+Y	16SR	16SR
	18	No. 007	YSR	YSR	GY	GY	MG	MG	89R+Y	93R+Y	32SR	36SR
18% Cr., 8% Ni.	15	No. 003	MG	MG	MG	MG	M	M	MG	MG	MG	MG
	12	No. 008	MG	MG	MG	MG	M	M	MG	MG	MG	MG
	20	No. 009	MG	MG	MG	MG	M	M	MG	MG	MG	MG
	14	No. 015	MG	MG	MG	MG	M	M	MG	MG	-	-

	Gage	Wire	Lafayette, Ind.		Ames, Iowa		Manhattan, Kans.		Ithaca, N.Y.		Santa Cruz, Calif.	
			13.00 Yr.	14.00 Yr.	12.93 Yr.	13.85 Yr.	12.92 Yr.	13.90 Yr.	12.93 Yr.	13.93 Yr.	12.83 Yr.	13.84 Yr.
Copper-covered wire	11	No. 021, 7.42-oz.	Gn	Gn	Br	Br	Br	Br	B	Br	Br	Br
	9	No. 022, 9.24-oz.	Gn	Gn	Br	Br	Br	Br	B	Br	Br	Br
Lead-coated Wires	6	No. 040, 1.40-oz.	93PHR	60PHR	3R	7R	5R*	GY	8R	9R	19SR	19SR
	9	No. 041, 1.48-oz.	93PHR	50PHR	39R	43R	20R	1SR	26R	11R	54SR	57SR
	11	No. 042, 1.68-oz.	81PHR	55PHR	24R	27R	Y	15R	11R	8R	22SR	22SR
	14-1/2	No. 043, 1.90-oz.	20PHR	25PHR	17R	25R	G	GY	6R	6R	26SR	29SR
Corrosion-Resistant Uncoated Wires:												
12-14% Cr., Cold drawn	12	No. 001	M	M	SY	SY	G	G	Y*	SY*	84SR	85SR
	9	No. 004	M	M	SY	SY	MG	MG	Y*	SY*	64SR	69SR
	117	No. 005	M	M	SY	SY	MG	MG	Y*	SY*	81SR	86SR
12-14% Cr., air quenched	13	No. 002	M	M	SY	SY	MG	MG	M	SY*	26SR	26SR
	10	No. 006	M	M	SY	SY	MG	M	M	SY*	25SR	25SR
	18	No. 007	M	M	SY	SY	MG	M	Y*	SY*	57SR	61SR
18% Cr., 8% Ni.	15	No. 003	M	M	M	M	M	M	M	M	MG	MG
	12	No. 008	M	M	M	M	M	M	M	M	MG	MG
	20	No. 009	M	M	SY	SY	M	M	M	M	MG	MG
	14	No. 015	M	M	SY	SY	M	M	M	M	-	-

TABLE II.—REPORT OF INSPECTIONS OF BARBED WIRE SPECIMENS.

Abbreviations and symbols used to designate appearance or states of corrosion: M = metallic; G = gray; Y = yellowed, or rust stained, but not showing actual rough rust of base metal; R = rust of base metal; PHR, PHY = rust or yellowed in pinholes; B, Br, Gn, respectively, signify corroded to a black, brown, or green appearance; D = dark (dirt or soot) to the exclusion of a better observation; MG = intermediate M and G; GY predominately gray but showing indication of Y; RB = rust on barbs; SR = superficial rust; asterisk (*) = speckled; R+Y = indistinguishable proportions of R and Y.

Coating Group	Average Coating, oz per sq ft of surface[a]	Wire[b]	State College, Pa.		Lafayette, Ind.		Ames, Iowa		Manhattan, Kans.		Ithaca, N. Y.		Santa Cruz, Calif.		College Station, Tex.		Davis, Calif.	
			11.42 yr	12.42 yr	13.00 yr	14.00 yr	12.92 yr	13.85 yr	12.92 yr	13.90 yr	12.93 yr	13.93 yr	12.83 yr	13.85 yr	12.84 yr	13.87 yr	12.84 yr	13.85 yr
0.20 to 0.30	0.30	No. C410....	100R	100R	100R	100R	100R	100R	80R	50R	100R	100R	40R	40R	90R	100R	10R	15R
	0.24	No. C411....	100R	100R	100R	100R	100R	100R	20R	50R	100R	100R	20R	40R	100R	100R	15R	15R
	0.29	No. C412....	100R	100R	100R	100R	100R	100R	GRB	20R	100R	100R	15R	20R	15R	20R	G	G
0.35 to 0.45	0.37	No. C421....	100R	100R	100R	100R	100R	100R	GY	GY	100R	100R	10R	15R	10R	15R	G	G
	0.47	No. C422....	100R	100R	100R	100R	50R	50R	GY	10R	100R	90R	10R	15R	12R	25R	G	G
	0.31	No. C420....	100R	100R	100R	100R	100R	100R	10R	20R	100R	100R	10R	15R	15R	25R	G	G
0.50 to 0.60	0.48	No. C430....	100R	100R	100R	100R	5R	5R	G	GY	100R	100R	10R	15R	5R	15R	G	G
	0.54	No. C431....	90R	90R	95R	100R	10R	10R	GRB	GY	50R	75R	15R	15R	10R	20R	G	G
	0.57	No. C432....	G	GRB	100R	100R	GRB	GRB	MG	30R	30R	90R	G	G	G	G	MG	MG
0.70 to 0.85	0.72	No. C441....	G	GRB	80R	95R	GRB	GRB	MG	MG	5R	30R	G	MG	G	G	MG	MG
	0.78	No. C442....	G	GRB	50R	95R	G	G	MG	MG	GRB	1RB	G	G	G	G	MG	MG
	0.74	No. C443....	GY	20R	95R	100R	GRB	GRB	G	G	20R	80R	G	G	G	G	MG	MG
0.80 to 1.00	0.97	No. C445....	GY	GY	Y	Y	G	G	MG	MG	GY	GY	G	G	G	G	MG	MG
	0.92	No. C446....	G	GY	20R	20R	G	G	MG	MG	GRB	1R	G	G	G	G	MG	MG
1.60 to 1.80	1.64	No. C450....	G	GY	10R	20R	G	G	G	MG	GY	GY	G	G	G	G	MG	MG
	1.50	No. C452....	G	G	G	G	G	G	MG	MG	GMG	G	G	G	G	G	MG	MG
	1.83	No. C451....	G	GRB	10R	15R	GRB	GRB	MG	MG	GRB	GRB	G	MG	G	G	MG	MG
Heavy Coating	1.99	No. C494....	G	G	G	G	G	G	MG	MG	M	M	G	G	G	G	MG	MG
	1.99	No. C494....	G	G	G	G	G	G	MG	MG	M	M	G	G	G	G	MG	MG
	2.00	No. C447....	G	GRB	15R	40R	GRB	GRB	MG	MG	GRB	GRB	G	G	G	G	MG	MG
Copper-covered (3.97 oz per sq ft)........		No. C025....	Gn	GnRB	Gn	Gn	.....	Br	Br	Br	....	....	BrRB	BrRB	Br	Br	BrRB	BrRB
		No. C025....	Gn	GnRB	....	....	Br	....	....	....	Gn	Gn	....	....	....	....	....	....
Lead-coated (1.83 oz per sq ft)...........		No. C045....	....	....	50PHR	15PHR	20R	20R	5PHY	15PHY	10R	10R	....	....	3R	4R	25SR	25SR
		No. C045....	....	....	50PHR	15PHR	20R	20R	....	....	....	....	30SR	30SR	3R	4R	....	....

[a] The zinc coating weight reported in this column is the average of all stripping test determinations made on samples of unfabricated wire, fence or barbed wire of the specific wire lot number.

[b] These barbed wire numbers do not always correspond to the unfabricated wire numbers as seen opposite:

Barbed Wire	Unfabricated Wire	Barbed Wire	Unfabricated Wire
No. C410	No. 410	No. C441	No. 442
No. C411	....	No. C442	No. 441
No. C412	No. 412	No. C443	No. 443
No. C421	....	No. C445	....
No. C422	No. 421	No. C446	....
No. C420	No. 423	No. C447	....
No. C430	No. 431	No. C450	....
No. C431	No. 430	No. C452	No. 450
No. C432	No. 432	No. C451	No. 451
		No. C494	....

TABLE III.—REPORT OF INSPECTIONS OF FARM FIELD FENCE SPECIMENS.

Abbreviations and symbols used to designate appearance or states of corrosion: M = metallic; G = gray; Y = yellowed, or rust stained, but not showing actual rough rust of base metal; R = rust of base metal; PHR, PHY = rust or yellowed in pinholes; B, Br, Gn, respectively, signify corroded to a black, brown or green appearance; D = dark dirt or soot) to the exclusion of a better observation; MG = intermediate between M and G; GY = predominately gray but showing indication of Y; SY or SR = superficial Y and superficial R, respectively; asterisk (*) = speckled appearance. Figures (as 5, 20, 50, 100) are percentages.

Coating Group	Average Coating, oz per sq ft of Surface[a]	Fence	State College, Pa.		Lafayette, Ind.		Ames, Iowa		Manhattan, Kans.		Ithaca, N. Y.		Santa Cruz, Calif.		College Sta., Tex.		Davis, Calif.	
			11.42 yr	12.42 yr	13.00 yr	14.00 yr	12.92 yr	13.85 yr	12.92 yr	13.90 yr	12.93 yr	13.93 yr	12.83 yr	13.84 yr	12.84 yr	13.87 yr	12.84 yr	13.85 yr
0.20 to 0.30 Avg 0.28	0.27	No. 512.....	100R	100R	100R	100R	100R	100R	50R	50R	100R	100R	25R	35R	98R	98R	40R	50R
	0.30	No. 410.....	100R	100R	100R	100R	100R	100R	50R	50R	100R	100R	50R	60R	50R	100R	25R	40R
	0.28	No. 411.....	100R	100R	100R	100R	100R	100R	Y	5R	100R	100R	10R	15R	15R	25R	G	1R
	0.29	No. 510.....	100R	100R	100R	100R	100R	100R	Y	15R	100R	100R	15R	25R	30R	40R	G	G
	0.29	No. 412.....	100R	100R	100R	100R	100R	100R	5R	20R	100R	100R	GY	30R	15R	20R	G	GY
	0.24	No. 511.....	100R	100R	100R	100R	100R	100R	45R	80R	100R	100R	30R	40R	95R	98R	50R	50R
0.25 to 0.35 Avg 0.30	0.25	No. 315.....	100R	100R	100R	100R	100R	100R	75R	50R	100R	100R	60R	75R	97R	98R	75R	90R
	0.28	No. 215.....	100R	100R	100R	100R	100R	100R	70R	50R	100R	100R	40R	45R	97R	50R	50R	75R
	0.38	No. 317.....	100R	100R	100R	100R	100R	100R	GY	GY	100R	100R	5R	10R	5R	10R	G	G
	0.32	No. 316.....	100R	100R	100R	100R	100R	100R	5R	10R	100R	100R	35R	10R	10R	15R	G	GY
	0.30	No. 216.....	100R	100R	100R	100R	100R	100R	GY	5R	100R	100R	5R	5R	10R	15R	G	GY
0.35 to 0.45 Avg 0.38	0.36	No. 520.....	100R	100R	100R	100R	100R	100R	GY	GY	95R	90R	10R	10R	35R	40R	G	G
	0.35	No. 422.....	100R	100R	100R	100R	100R	100R	GY	10R	95R	90R	GY	25R	20R	30R	G	G
	0.31	No. 423.....	100R	100R	100R	100R	100R	90R	GY	GY	95R	90R	10R	10R	15R	25R	G	G
	0.44	No. 522.....	100R	100R	100R	100R	100R	50R	Y	G	85R	90R	8R	10R	10R	15R	G	G
	0.38	No. 521.....	100R	100R	100R	100R	100R	50R	Y	10R	90R	90R	10R	25R	25R	30R	3R	G
	0.47	No. 421.....	100R	100R	100R	100R	25R	25R	GY	GY	80R	90R	10R	20R	15R	20R	G	G
0.45 to 0.55 Avg 0.46	0.44	No. 325.....	100R	100R	100R	100R	10R	5R	GY	GY	75R	80R	15R	15R	10R	15R	G	G
	0.50	No. 228.....	100R	100R	100R	100R	30R	30R	GY	GY	70R	75R	25R	30R	10R	15R	G	G
	0.51	No. 227.....	100R	100R	100R	100R	50R	70R	GY	GY	70R	75R	20R	25R	5R	15R	G	G
	0.38	No. 326.....	100R	100R	100R	100R	80R	50R	GY	GY	85R	90R	8R	12R	5R	10R	G	G
	0.47	No. 327.....	100R	100R	100R	100R	G	G	GY	G	80R	80R	10R	10R	3R	5R	G	G
	0.45	No. 225.....	100R	100R	100R	100R	5R	5R	G	GY	70R	70R	10R	10R	5R	10R	G	G
0.50 to 0.60 Avg 0.54	0.61	No. 530....	G	30R	80R	80R	G	G	Y	MG	20R	20R	MG	MG	G	G	MG	MG
	0.57	No. 432....	G	G	80R	80R	G	G	MG	MG	1R	25R	MG	MG	G	G	MG	MG
	0.60	No. 531....	100R	100R	100R	100R	50R	20R	G	G	50R	60R	MG	GY	G	G	MG	MG
	0.48	No. 431....	100R	100R	100R	100R	10R	10R	G	GY	75R	100R	GY	GY	10R	20R	G	G
	0.54	No. 430....	100R	100R	100R	100R	10R	10R	GY	GY	80R	80R	10R	10R	10R	15R	G	G
	0.49	No. 532....	100R	100R	100R	100R	G	G	Y	GY	75R	60R	GY	20R	5R	10R	G	G
	0.49	No. 433....	100R	100R	100R	100R	GY	G	GY	GY	75R	80R	GY	GY	10R	15R	G	G
0.60 to 0.70 Avg. 0.64	0.69	No. 338....	G	GY	30R	25R	G	G	MG	MG	5R	40R	MG	MG	G	G	MG	MG
	0.68	No. 236....	G	G	G	10R	G	G	MG	MG	MG	MG	MG	MG	G	G	MG	MG
	0.61	No. 335....	50R	95R	85R	85R	G	G	G	G	35R	40R	G	G	GG	G	MG	MG
	0.63	No. 237....	50R	70R	75R	75R	G	G	G	G	50R	70R	MG	MG	1R	1R	G	MG
	0.57	No. 337....	100R	100R	80R	80R	G	G	GY	GY	60R	70R	5R	10R	10R	15R	G	G
	0.67	No. 235....	90R	90R	90R	90R	G	G	GY	GY	70R	75R	10R	10R	10R	10R	G	G
	0.66	No. 336....	100R	90R	80R	85R	G	G	GY	GY	45R	60R	10R	15R	10R	15R	G	G
	0.60	No. 238....	70R	90R	80R	85R	G	G	GY	GY	60R	60R	15R	20R	5R	10R	G	G

0.70	0.70	No. 540...	G	G	35R	40R	G	G	MG	MG	5R	10R	MG	MG	G	G	MG	MG
to	0.72	No. 442...	G	G	G	G	G	G	MG	MG	MG	MG	MG	MG	G	G	MG	MG
0.85	0.79	No. 543...	GY	40R	80R	90R	G	G	MG	MG	10R	35R	MG	G	G	G	MG	MG
Avg	0.78	No. 441...	GY	5R	20R	80R	G	G	MG	MG	1R	50R	G	G	G	G	MG	MG
0.74	0.70	No. 542...	GY	40R	75R	80R	G	G	MG	G	25R	25R	G	G	G	G	MG	MG
	0.74	No. 443...	GY	50R	50R	75R	G	G	MG	MG	50R	50R	MG	MG	G	G	MG	MG
	0.76	No. 541...	GY	40R	75R	85R	G	G	MG	MG	10R	30R	G	G	G	G	MG	MG
	0.76	No. 440...	GY	10R	75R	75R	G	G	MG	MG	10R	100R	G	G	G	G	MG	MG
0.80	1.05	No. 348...	G	G	G	G	G	G	MG	MG	MG	MG	MG	MG	G	G	MG	MG
to	0.81	No. 248...	G	G	G	G	G	G	MG	MG	M	M	MG	MG	G	G	MG	MG
1.00	0.88	No. 347...	G	GY	10R	30R	G	G	MG	G	GY	10R	15R	15R	G	G	MG	MG
Avg	0.95	No. 246...	G	GY	10R	15R	G	G	MG	MG	GY	GY	G	G	G	G	G	MG
0.90	0.88	No. 346...	Y	GY	30R	55R	G	G	G	G	1R	10R	G	G	G	G	MG	MG
	0.81	No. 245...	Y	5R	60R	70R	G	G	MG	G	1R	15R	MG	MG	G	G	MG	MG
	0.84	No. 345...	Y	30R	30R	60R	G	G	GY	GY	10R	10R	2R	5R	5R	7R	G	G
	0.96	No. 247...	Y	GY	30R	40R	G	G	GY	GY	5R	30R	5R	5R	5R	7R	G	G
1.60	1.75	No. 350...	G	G	G	G	G	G	MG	MG	M	MG	MG	MG	G	G	MG	MG
to	1.69	No. 252...	G	G	G	G	G	G	MG	MG	M	M	MG	MG	G	G	MG	MG
1.80	1.75	No. 352...	G	G	G	G	G	G	MG	MG	M	MG	G	G	G	G	MG	G
Avg	2.80	No. 397...	G	G	G	G	G	G	MG	MG	M	M	MG	MG	G	G	MG	MG
2.76	2.72	No. 298...	G	G	G	G	G	G	MG	MG	M	M	MG	MG	G	G	MG	MG
12-14% Cr, cold drawn		No. 001...	MG	MG	M	M	SY	SY	MG*	MG*	M*	MG*	75SR	75SR	15R	20R	35SR	40R
12-14% Cr, air-quenched		No. 002...	MG	MG	M	M	SY	SY	MG	MG*	M*	MG*	35SR	35SR	5SR	5R	25SR	30SR
18% Cr, 8% Ni...		No. 003...	M	M	M	M	M	M	M	M	M	M	MG	MG	MSY*	M	MG	MG
Copper-covered (7.42 oz per sq ft).....		No. 020...	Gn	Gn	Gn	Gn	Br	Br	Br	Br	Gn	Gn	Br	Br	Br	Br	Br	Br
Lead-coated (1.48 oz per sq ft).........		No. 040...	100Y+R	100Y+Y	50PHR	15PHR	25R	25R	15R	20R	5R	10R	40SR	40SR	15R	15R	40SR	40R

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[a] The zinc coating weight reported in this column is the average of all stripping test determinations made on samples of unfabricated wire, fence or barbed wire of the specific wire lot number.

TABLE IV.—REPORT OF INSPECTIONS OF CHAIN LINK SPECIMENS.

Abbreviations and symbols used to designate appearance or states of corrosion: M = metallic; G = gray, Y = yellowed, or rust stained, but not showing actual rough rust of base metal; R = rust of base metal; PHR, PHY = rust or yellowed in pinholes; B, Br, Gn, respectively, signify corroded to a black, brown, or green appearance; D = dark (dirt or soot) to the exclusion of a better observation; MG = intermediate between M and G; GY = predominately gray but showing indication of Y; R+Y = indistinguishable proportions of R and Y; SR = superficial rust. Figures (as 5, 20, 50, 100) are percentages.

All 200 series wires are 9 gage. Lead-coated wire, 9 gage. Corrosion-resistant steel, 14 gage.

All 100 series wires are 6 gage. Copper-covered wire 0.128d.

Zinc Coating oz per sq ft of surface, by stripping test	Fence	Sandy Hook, N. J.		Bridgeport, Conn.		State College, Pa.		Lafayette, Ind.		Ithaca, N. Y.		Santa Cruz, Calif.		Davis, Calif.	
		12.44 yr	13.44 yr	12.45 yr	13.44 yr	11.42 yr	12.42 yr	13.00 yr	14.00 yr	12.93 yr	13.93 yr	12.83 yr	13.84 yr	12.84 yr	13.85 yr
0.29	No. E280[a]	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	15R	25R	15R	15R
0.52	No. E281	100R	100R	100R	100R	80R	90R	100R	100R	90R	90R	5R	8R	5R	5R
0.37	No. E282	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	5R	10R	G	G
1.22	No. E185	100R	100R	100R	100R	Y	GY	GY	GY	GY	GY	MG	MG	MG	MG
1.13	No. E285	100R	100R	100R	100R	Y	GY	10R	GY	GY	GY	MG	MG	G	MG
2.92	No. E290	G	G	G	G	G	G	G	G	M	M	MG	MG	MG	MG
2.09	No. E291	G	G	G	G	G	G	G	G	MG	MG	MG	MG	MG	MG
1.60	No. H280[a]	100R	100R	90R	100R	G	GY	5R	5R	GY	G	G	G	MG	MG
1.83	No. H185	100R	100R	90R	90R	GY	GY	GY	GY	GY	GY	MG	MG	MG	MG
1.60	No. H186	100R	100R	90R	100R	G	GY	GY	GY	GY	GY	G	MG	MG	MG
1.61	No. H187	100R	70R	65R	70R	G	GY	GY	GY	G	GY	G	G	MG	MG
1.55	No. H188	100R	100R	90R	100R	GY	GY	GY	GY	GY	GY	MG	MG	MG	MG
1.36	No. H285	100R	100R	90R	100R	GY	GY	GY	GY	G	GY	G	G	G	MG
1.97	No. H286	100R	100R	70R	100R	GY	GY	GY	GY	GY	GY	MG	MG	MG	MG
1.46	No. H287	100R	100R	80R	100R	G	GY	GY	GY	GY	GY	MG	MG	MG	MG
1.74	No. H288	100R	100R	70R	100R	G	GY	GY	GY	G	GY	G	G	G	MG
1.40	No. H289[b]	5R	30R	GY	10R	G	GY	G	G	MG	MG	MG	MG	MG	MG
4.21	No. H290	GY	GY	GY	Y	G	G	G	GY	GY	G	MG	MG	MG	MG
Lead-coated L050 (1.75 oz per sq ft)		50R+Y	50R+Y	Y	Y	100R+Y	100R+Y	100PHR	100R+Y	10R	100R+Y	35SR	35SR	35SR	35SR
Copper-covered K030 (8.52 oz per sq ft)		Gn	Gn	Gn	Gn	Gn	Gn	Gn	Gn	Br	B	Br	Br	Br	Br
Corrosion-resistant steel J011		G	G	MG	MG	MG	M	M	M	M	M	MG	MG	MG	MG

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[a] E numbers, coating applied before weaving. H numbers, coating applied after weaving.

[b] While this sample has an H number, its apperance indicates that it was galvanized before weaving.

TABLE V.—REPORT OF INSPECTIONS OF WIRE STRAND SPECIMENS.

Abbreviations and symbols used to designate appearance or states of corrosion: M = metallic; G = gray; Y = yellowed, or rust stained, but not showing actual rough rust of base metal; R = rust of base metal; PHR, PHY = rust or yellowed in pinholes; B, Br, Gn, respectively, signify corroded to a black, brown or green appearance; D = dark (dirt or soot) to the exclusion of a better observation; MG = intermediate between M and G; GY = predominately gray but showing indication of Y; R+Y = indistinguishable proportions of R and Y.

Zinc Coating, oz per sq ft of surface	Strand	Sandy Hook, N. J.		Bridgeport, Conn.		State College, Pa.		Lafayette, Ind.		Ames, Iowa	
		12.44 yr	13.44 yr	12.45 yr	13.44 yr	12.30 yr	13.30 yr	13.00 yr	14.00 yr	12.92 yr	13.85 yr
0.69	No. D345....	25R	40R	75R	75R	G	G	G	G	G	G
0.88	No. D346....	25R	40R	25R	40R	G	GY	GY	GY	G	G
0.87	No. D347....	55R	60R	25R	40R	G	GY	GY	5R	G	G
0.93	No. D348....	50R	60R	45R	50R	G	G	G	G	G	G
1.10	No. D360....	G	2R	GY	2R	G	G	G	G	G	G
1.17	No. D361....	10R	15R	GY	2R	G	GY	GY	GY	G	G
1.03	No. D362....	50R	60R	10R	50R	GY	GY	GY	GY	G	G
1.51	No. D365....	Y	Y	GY	1R	G	GY	GY	GY	G	G
1.39	No. D366....	G	1R	GY	GY	G	G	G	G	G	G
1.32	No. D367....	10R	20R	Y	10R	GY	GY	Y	Y	G	G
1.71	No. D371....	G	G	GY	GY	G	G	G	G	G	G
2.47	No. D375....	G	G	GY	GY	G	G	G	G	G	G
Lead Coating (1.96 oz per sq ft).......	No. D045....	100R+Y	100R+Y	5R	10R	100R+Y	100R+Y	50PHR	50PHR	40R	40R

Zinc Coating, oz per sq ft of surface	Strand	Manhattan, Kans.		Ithaca, N. Y.		Santa Cruz, Calif.		College Station, Tex.		Davis, Calif.	
		12.92 yr	13.90 yr	12.93 yr	13.93 yr	12.83 yr	13.84 yr	12.84 yr	13.87 yr	12.84 yr	13.85 yr
0.69	No. D345....	G	MG	G	1R	G	1R	G	G	G	G
0.88	No. D346....	G	MG	GY	GY	MG	MG	G	G	MG	MG
0.87	No. D347....	G	GY	GY	GY	G	G	G	G	G	G
0.93	No. D348....	MG	MG	MG	MG	MG	MG	G	G	G	G
1.10	No. D360....	MG	MG	MG	MG	G	MG	G	G	G	MG
1.17	No. D361....	MG	MG	GY	GY	G	G	G	G	G	G
1.03	No. D362....	MG	MG	GY	GY	MG	MG	G	G	G	G
1.51	No. D365....	G	GY	GY	GY	G	G	G	G	G	G
1.39	No. D366....	MG	MG	MG	G	G	MG	G	G	G	G
1.32	No. D367....	MG	MG	GY	GY	MG	MG	G	G	G	G
1.71	No. D371....	MG	MG	MG	MG	MG	MG	G	G	G	MG
2.47	No. D375....	MG	MG	MG	MG	MG	G	G	G	G	G
Lead Coating (1.96 oz per sq ft).......	No. D045....	10R	10R	15R	20R	60SR	60SR	10R	15R	10R	10R

Findings from Visual Examination for Corrosion at the Test Plots:

At the time of inspection, records are made of the appearance of each specimen. Prior to the time of rusting the specimens are noted as appearing "metallic," "gray," "yellow," etc. After rusting has begun, estimates are made of the percentage of area rusted on each specimen.

The condensed and summarized inspection records of corrosion through 1950 are shown in Tables A through F. The detailed inspection records collected in 1949 and 1950 are presented in Table I (Plate I) for unfabricated wire, Table II for barbed wire, Table III for farm field fence, Table IV for chain link fence, and Table V for wire strand. These tables are similar in form to the tables in previous reports. Detailed records for the Pittsburgh, Pa., site are not included in the tables because, in general, everything exposed at Pittsburgh has already been reported as 100 per cent rusted, or is so covered with soot that a good observation is not possible.

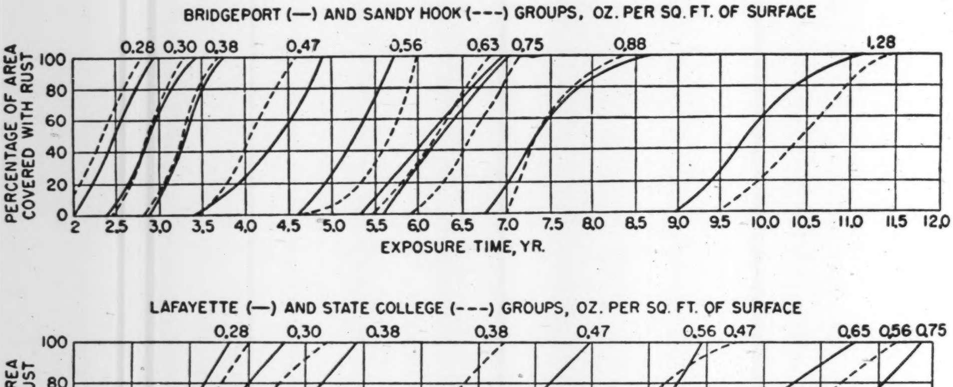

FIG. 1.—Progressive Development of Rust on Zinc-Coated Unfabricated Wire. (Coating Weight Groups in which all of the samples are rusted.)

The corrosion performance of any individual specimen of barbed wire or of farm field fence listed in Tables II and III may be directly compared with the performance of specific unfabricated wire specimens listed in Table I (Plate I). The Reference Information Table (Plate II) in this respect shows the lot numbers of wires used in fabricating all farm field fence and barbed wire samples.

In Fig. 1 is shown the progressive development of rust on zinc-coated, unfabricated wire specimens for all groups which have become 100 per cent rusted and for all groups of wire which, while not 100 per cent rusted, show some rust on each specimen of the group. In this Fig. 1 are included groups from Sandy Hook, Bridgeport, Lafayette, State College, Ithaca, and Ames sites. The data from all groups at the Pittsburgh site were reported in 1947.[2] Specimens at other locations have not yet rusted to the extent that the development of rust can be shown graphically for these sites.

TABLE G.—NUMBER OF TENSILE STRENGTH SPECIMENS REMOVED FOR TESTS.

Exposure Site	Number of Wires Removed in 1949 and 1950				Wires Removed Prior to 1949	Total Removed to Date
	Bare Steel	Zinc-Coated	Lead-Coated	Other Types		
Pittsburgh, Pa.	12	74	4	12	658	760
Sandy Hook, N. J.	0	18	0	0	362	380
Bridgeport, Conn.	4	26	0	0	307	337
State College, Pa.	0	0	0	0	118	118
All Other Sites	0	0	0	0	415	415
Total	16	118	4	12	1860	2010

Findings From Specimens Removed for Tension Tests:

Tension tests are made on the unfabricated wire specimens which were exposed in groups of seven comparison samples taken from the same lot of wire and supposedly alike. An attempt is made to remove and test the first specimen of each group when it has lost 5 to 10 per cent of its strength, and the seventh specimen when it has lost some 75 per cent of its strength. The specimens from the Pittsburgh test site have been removed at an accelerated rate because the test site is to be abandoned soon. There were originally about 840 tensile strength specimens exposed at each site. The number of tensile strength specimens removed to date is shown in Table G. The results of tensile strength and elongation tests on the 1860 wires removed prior to 1949 have been published in previous reports. The results of tests on specimens removed in 1949 and 1950 are shown in Table VI.

TABLE H.—LOSS OF TENSILE STRENGTH—UNCOATED STEEL WIRES.

Exposure Site	Loss in Tensile Strength, per cent per yr. Exposure				
	No. 14½ Gage	No. 12½ Gage	No. 11 Gage	No. 9 Gage	No. 6 Gage
Pittsburgh, Pa.	14	10	7	5	3
Sandy Hook, N. J.	10	6	4	3	2
Bridgeport, Conn.	6	3	2	2	1
State College, Pa.	5	2	2	2	1
Lafayette, Ind.	4	2	2	2	1
Santa Cruz, Calif.	3	2	1	1	.
Ithaca, N. Y.	3	2	1	1	.
Ames, Iowa	3	2	1	1	.
College Sta., Texas	2	1	1	.	.
Davis, Calif.	2	1	.	.	.
Manhattan, Kans.	1	.	.	.	.

On the basis of tests to date the loss in tensile strength by uncoated steel wires expressed in per cent per year of exposure for the various gages of wires is as shown in Table H. Although these data are for uncoated wire, other data such as those in Table J, have indicated that after the coating has gone, the rate of loss is the same whether wires were previously coated or not. In Table J is shown the tensile strength data obtained from all the uncoated and zinc-coated mild steel specimens exposed at Pittsburgh, Pa.

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[2] Report of Wire Inspection Committee on Field Tests of Wire and Wire Products, *Proceedings*, Am. Soc. Testing Mats., Vol. 47, p. 120 (1947).

TABLE J - LOSS OF TENSILE STRENGTH SHOWN BY STEEL WIRES AFTER VARIOUS PERIODS OF EXPOSURE

AT PITTSBURGH, PA.

		First Test			Second Test			Third Test			Fourth Test			Fifth Test			Sixth Test			Seventh Test		
Wire Lot*	Exposure Until First Rust Yrs.	Exp. Since 1R Yr.	Tensile Strength Loss %	Tensile Strength Loss %/Yr.	Exp. Since 1R Yr.	Tensile Strength Loss %	Tensile Strength Loss %/Yr.	Exp. Since 1R Yr.	Tensile Strength Loss %	Tensile Strength Loss %/Yr.	Exp. Since 1R Yr.	Tensile Strength Loss %	Tensile Strength Loss %/Yr.	Exp. Since 1R Yr.	Tensile Strength Loss %	Tensile Strength Loss %/Yr.	Exp. Since 1R Yr.	Tensile Strength Loss %	Tensile Strength Loss %/Yr.	Exp. Since 1R Yr.	Tensile Strength Loss %	Tensile Strength Loss %/Yr.
No. +501[a]	0	1.48	36.0	24	1.94	39.9	20	2.45	46.0	19	2.93	49.0	17	3.48	55.0	16	4.50	61.0	13	5.48	87.0	16
No. +506	0	1.48	32.7	22	1.94	35.5	18	2.45	37.5	15	2.93	45.2	15	3.48	48.1	14	4.50	57.8	13	5.48	82.7	15
No. +507	0	1.48	31.1	21	1.94	36.1	18	2.45	37.8	15	2.93	47.1	16	3.48	52.9	15	4.50	59.6	13	5.48	84.9	15
No. 510	1.20	.28	0	0	.74	10.0	13	1.25	14.0	11	1.73	25.0	15	2.28	31.0	14	3.72	46.0	12	4.75	65.0	14
No. 511	0.60	.88	26.3	30	1.34	28.1	21	1.85	42.5	23	2.33	38.6	17	2.88	43.8	15	4.32	59.6	14	5.35	79.0	14
No. 512	0.50	.98	26.1	27	1.44	27.7	19	1.95	30.8	16	2.43	41.5	17	2.98	44.6	15	4.42	49.2	11	5.45	83.0	15
No. 520	1.20	.28	0	0	.74	1.8	2	3.30	32.1	10	4.28	58.9	14	4.75	55.4	12	4.75	55.4	12	5.75	71.5	12
No. 521	1.75	0	-3.7	0	.20	-5.5	0	2.75	33.3	12	3.73	59.3	16	4.20	55.6	13	4.20	55.6	13	5.20	75.9	14
No. 522	1.45	0	-2.0	0	.50	5.0	10	3.05	40.0	13	4.03	65.0	16	4.50	64.0	14	4.50	64.0	14	5.50	79.7	14
No. 530	1.20	.28	-1.3	0	.74	-4.5	0	3.72	50.0	13	4.28	72.6	17	4.75	62.9	13	5.30	53.3	13	5.75	80.7	14
No. 531	1.40	.08	8.0	0	.54	15.0	28	3.52	50.0	14	4.08	63.0	15	4.55	66.0	15	5.10	82.0	16	5.55	79.8	14
No. 540	1.85	.10	-6.8	0	3.07	53.4	18	3.63	68.3	19	4.10	61.7	15	4.60	71.7	16	5.10	78.3	15	5.65	80.0	14
No. 541	2.60	0	-1.5	0	2.32	32.9.	14	2.88	65.7	23	3.35	57.2	17	3.90	68.5	17	4.35	72.9	17	4.90	75.7	15.
No. 542	1.65	.30	0	0	3.27	41.2	13	3.83	57.8	15	4.30	59.8	14	4.80	65.7	14	5.30	71.6	14	5.85	77.4	13
No. 543	1.85	.10	-1.7	0	3.07	35.0	12	3.63	68.3	19	4.10	55.0	13	4.60	55.0	12	5.10	73.3	14	5.65	75.0	13
No. 551	4.50	.42	4.6	11	2.00	46.3	23	2.45	47.2	19	3.00	63.8	21	3.40	77.8	23	3.40	77.8	23	-	-	-
No. 552	4.90	0	-1.0	0	1.60	35.5	21	2.05	42.0	20	2.60	45.1	17	3.00	64.5	21	3.55	71.0	20	4.5	77.4	17
No. +400[a]	0	1.48	25.0	17	1.94	23.0	12	2.45	25.0	10	2.93	32.3	11	3.48	32.3	9	4.92	43.8	9	5.95	54.2	9
No. +401[a]	0	1.48	28.9	20	1.94	25.5	13	2.45	31.1	13	2.93	38.0	13	3.48	41.2	12	4.92	50.0	10	5.95	65.6	10
No. +405	0	1.48	23.6	16	1.94	23.7	12	2.45	23.7	10	2.93	33.0	11	3.48	33.0	10	4.92	43.5	9	5.95	56.8	9
No. +406	0	1.48	21.3	14	1.94	21.3	11	2.45	22.3	9	2.92	27.7	9	3.48	28.7	8	4.92	37.2	7	5.95	44.7	7
No. 410	0.65	.83	18.0	22	1.29	18.8	15	2.28	28.1	12	2.83	30.7	11	4.27	45.7	11	4.83	64.9	13	5.30	51.7	10
No. 411	0.85	.63	7.3	12	1.09	9.7	9	2.08	16.3	8	2.63	26.8	10	4.07	31.8	8	4.63	41.5	9	5.10	45.5	9
No. 412	1.25	.23	4.8	21	.69	8.5	12	1.68	17.5	10	2.23	19.3	9	3.67	54.2	15	4.23	-	-	4.70	40.4	9
No. 421	1.60	0	4.3	0	.34	3.2	10	2.90	25.5	9	3.88	40.4	10	4.35	28.6	7	5.35	49.8	9	5.35	55.2	10
No. 422	1.45	0	8.0	0	.50	5.0	10	3.05	23.0	8	4.03	40.0	10	4.50	34.0	8	-	-	-	-	-	-
No. 423	1.25	.23	17.5	-	.70	17.5	25	3.25	25.0	8	4.23	41.2	10	4.70	40.0	9	-	-	-	-	-	-
No. 430	2.60	0	-1.3	0	0	-4.5	0	2.32	15.4	7	2.88	35.3	12	3.35	38.5	11	4.35	50.0	12	5.30	56.5	11
No. 431	1.60	0	6.8	0	.34	11.4	33	3.32	23.9	7	3.88	41.5	11	4.35	39.8	9	5.35	53.4	10	6.30	59.1	9
No. 432	1.60	0	-2.0	0	.34	9.8	29	3.32	-	-	3.88	38.2	10	4.35	42.2	10	5.35	52.0	10	6.30	62.7	10
No. 440	2.60	0	-6.8	0	2.32	31.4	14	2.88	39.8	14	3.35	41.6	12	3.90	48.3	12	4.90	52.5	11	5.30	63.5	12
No. 441	1.90	.55	-5.8	0	3.02	23.1	8	3.58	41.3	12	4.05	42.4	10	4.60	49.1	11	5.60	60.5	11	6.00	63.5	11
No. 442	2.00	.45	7.5	17	2.92	10.6	3	3.48	45.7	13	3.95	46.8	12	4.50	52.2	11	5.50	57.4	10	5.90	63.9	11
No. 443	1.90	.55	-1.3	0	3.02	22.0	7	3.58	38.0	11	4.05	41.4	10	4.60	46.0	10	5.60	58.0	10	6.00	60.6	10
No. 444	2.70	0	0	0	2.22	21.1	10	2.78	37.7	14	3.25	43.8	13	3.80	43.0	11	4.80	56.1	12	5.20	57.0	11
No. 495	1.90	0.55	-0.7	0	3.02	23.4	8	3.58	38.0	11	4.05	38.0	11	4.60	44.7	10	5.60	52.0	9	6.00	57.7	10
No. 450	2.95	2.53	22.6	8	3.50	24.4	7	4.00	40.9	10	4.55	44.7	10	5.00	51.8	10	5.50	57.4	10	6.5	65.3	10
No. 451	4.50	0.98	21.6	21	2.00	23.6	12	2.45	34.3	14	3.00	34.3	11	3.40	43.1	13	3.95	47.1	12	4.9	50.0	10
No. +300[a]	0	1.48	20.1	14	1.94	18.2	9	2.45	19.6	8	2.93	24.3	8	3.48	23.6	7	5.95	40.5	7	10.47	63.5	6
No. +302[a]	0	1.48	25.5	17	1.94	25.5	12	2.45	25.4	10	2.93	31.3	11	3.48	31.8	9	5.95	48.1	8	10.47	74.5	7
No. +305	0	1.48	20.0	14	1.94	15.2	8	2.45	18.9	8	3.48	24.4	7	5.95	39.2	7	7.50	50.2	7	10.47	64.0	6
No. +306	0	1.48	17.0	12	1.94	14.8	8	2.45	15.5	6	3.48	19.7	6	5.95	36.0	6	7.50	46.8	6	10.47	58.5	6

No. 315	0.45	1.03	14.4	14	1.50	9.0	6	3.03	18.3	6	5.03	36.7	7	6.00	36.6	6	9.0	61.7	7	11.0	73.3	7
No. 316	1.20	.28	2.2	8	.74	5.7	8	2.28	10.1	4	4.28	24.6	6	5.30	28.0	5	8.2	53.6	7	10.3	64.7	6
No. 317	1.45	.49	5.6	11	1.48	13.8	9	2.03	15.1	8	4.03	32.1	8	5.00	34.6	7	8.0	40.8	5	-	-	-
No. 325	1.75	0.00	1.4	0	.20	-2.1	0	3.17	9.6	3	4.70	25.3	5	5.75	32.2	6	7.7	53.4	7	9.7	61.5	
No. 326[a]....	1.40	.08	4.3	-	.54	12.9	25	3.52	16.5	5	5.10	47.2	9	6.10	57.8	9	8.0	74.3	9	10.1	91.4	9
No. 327	1.80	0	3.5	0	.14	2.3	16	3.12	20.3	7	4.70	29.9	6	5.70	39.2	7	7.6	54.8	7	9.7	70.3	8
No. 329	1.85	0	1.4	0	.10	-4.2	0	3.07	16.7	5	4.60	30.6	7	5.65	40.3	7	7.6	54.8	7	9.7	68.7	7
No. 335	1.60	.95	5.8	6	3.32	18.7	6	3.88	30.3	8	4.90	29.3	6	5.90	39.2	7	7.8	53.8	7	9.9	68.3	7
No. 336	2.75	0	0	0	2.17	5.9	3	2.73	31.6	12	3.70	27.0	7	4.75	34.8	7	6.7	58.5	9	8.7	67.2	
No. 337	2.50	0	0	0	2.42	15.1	6	2.98	34.3	12	4.00	32.0	8	5.00	40.7	8	7.0	54.2	8	9.0	70.4	8
No. 338	2.00	.45	2.0	4	2.92	14.2	5	3.48	29.4	8	4.50	28.4	6	5.50	39.1	7	7.4	50.0	7	9.5	67.3	7
No. 345	2.70	2.22	21.9	10	2.78	23.0	8	3.80	23.6	6	4.80	34.8	7	6.7	54.5	8	8.8	66.8	8	9.8	74.8	8
No. 346	2.55	2.37	14.0	6	2.93	27.6	9	3.95	29.0	7	4.95	37.8	8	6.9	50.0	7	8.9	70.0	8	10.0	74.8	7
No. 347	2.65	2.27	23.2	10	2.83	25.0	9	3.80	32.0	8	4.85	38.9	8	6.8	46.5	7	8.8	68.5	8	9.9	76.8	8
No. 348	3.20	1.72	16.9	10	2.28	24.0	10	3.30	14.8	5	4.30	35.7	8	6.2	46.4	7	8.3	65.8	8	9.3	65.8	7
No. 350	5.10	.38	7.0	21	1.40	13.3	10	2.40	22.6	9	3.55	25.8	7	5.4	39.3	7	7.4	53.2	7	8.2	67.2	8
No. 351	2.90	2.58	13.9	5	3.50	13.9	4	4.60	25.8	6	5.55	31.5	6	7.6	43.4	6	9.6	51.8	6	10.4	63.4	6
No. 352	3.90	1.58	7.8	5	2.50	16.0	6	3.60	27.6	8	4.55	32.4	7	6.6	39.3	6	8.6	56.3	7	9.4	63.4	7
No. 397	7.50	1.00	14.9	15	1.90	22.9	12	3.0	28.7	9	4.0	34.6	8	5.0	38.3	8	5.8	36.7	6	-	-	-
No. +200[a]....	0	1.48	20.5	14	1.94	19.2	10	2.93	25.8	9	3.48	26.8	8	5.95	35.9	6	11.5	69.8	6	12.5	72.7	6
No. +202[a]....	0	1.48	18.8	13	1.94	18.0	9	2.93	18.0	6	3.48	23.2	7	5.95	31.7	5	11.5	56.4	5	12.5	59.7	5
No. +205	0	1.48	16.2	11	1.94	11.4	6	2.93	18.8	6	3.48	20.3	6	5.95	29.3	5	11.5	56.4	5	12.5	64.0	5
No. +206	0	1.48	15.5	11	1.94	16.5	8	2.93	17.9	6	3.48	17.9	5	5.95	27.1	5	11.5	53.4	5	12.5	56.4	5
No. 215	0.60	.88	9.9	11	1.34	9.9	7	2.88	12.8	4	4.88	27.7	6	6.90	33.1	5	10.9	34.5	5	11.9	59.1	5
No. 216	1.30	.18	1.8	10	.64	6.1	10	2.18	11.0	5	4.18	23.3	5	6.20	27.5	4	10.2	47.8	5	11.2	50.6	5
No. 217	1.05	.43	1.9	4	.90	0	0	2.43	-	-	4.43	19.0	4	6.45	18.0	3	10.4	36.6	4	11.4	42.1	4
No. 282	1.45	.50	0.8	2	1.48	11.0	7	3.05	14.4	5	4.03	23.1	6	4.50	22.0	6	6.5	34.1	5	-	-	-
No. 225	1.90	0	1.2	0	.04	0.9	0	3.02	15.1	5	4.60	18.5	4	9.6	43.6	5	10.6	46.1	4	11.4	51.4	5
No. 226	1.90	0	3.5	0	.04	-1.7	0	3.02	16.5	5	4.60	17.4	4	9.6	38.2	4	10.6	40.8	4	11.4	46.5	4
No. 227	2.50	0	3.0	0	0	4.3	0	2.42	20.0	8	4.00	-	-	9.0	46.4	5	10.0	46.7	4	10.8	53.4	5
No. 228	1.60	0	0	0	.34	-0.5	0	3.32	14.8	4	4.90	15.7	3	9.9	38.4	4	10.9	38.9	4	11.7	47.7	4
No. 235	2.60	0.43	2.4	6	2.32	30.0	13	2.88	21.4	7	4.90	23.5	5	8.9	55.0	6	9.9	56.6	6	10.7	48.2	5
No. 236	2.00	0.43	0	0	2.92	5.9	2	3.48	12.6	4	5.50	18.4	3	9.5	37.4	4	10.5	40.6	4	11.3	43.7	4
No. 237	1.80	..04	-2.8	0	3.12	15.9	5	3.68	19.5	5	5.70	26.9	5	9.7	44.2	5	10.7	43.5	4	11.5	51.3	4
No. 238	2.50	.04	-2.1	0	2.42	11.5	5	2.98	26.2	9	5.00	26.9	5	9.0	43.4	5	10.0	45.7	5	10.8	50.3	5
No. 239	2.60	0	-0.9	0	2.32	11.6	5	2.88	20.1	7	4.90	24.1	5	8.9	38.0	4	9.9	39.3	4	10.7	45.5	4
No. 245	2.40	2.52	14.6	6	3.08	18.8	6	4.10	19.4	5	7.0	33.1	5	9.1	47.2	5	10.1	48.1	5	-	-	-
No. 246	2.90	2.02	8.8	4	2.58	15.6	6	3.60	14.7	4	6.5	30.3	5	7.6	35.7	5	8.6	46.3	5	9.6	48.4	5
No. 247	2.90	2.02	12.3	6	2.58	23.5	9	3.60	24.0	7	6.5	27.7	4	8.6	47.8	6	9.6	45.0	5	9.6	50.9	5
No. 248	2.10	2.82	14.1	5	3.38	19.3	6	4.40	14.1	3	7.3	31.4	4	9.4	44.8	5	10.4	42.8	4	11.2	50.4	5
No. 296	2.50	2.32	10.8	5	2.98	15.3	5	4.00	15.6	4	6.9	29.3	4	9.0	40.8	5	10.0	38.2	4	10.8	44.2	4
No. 801	3.50	1.42	22.5	16	1.98	30.2	15	3.00	33.3	11	4.95	38.7	8	7.00	48.4	6	9.0	48.7	5	9.8	52.5	5
No. 250	3.90	1.58	7.2	4	2.50	3.6	2	4.55	18.5	4	6.6	29.7	5	8.6	37.3	4	9.4	40.2	4	10.6	43.1	4
No. 251	3.90	1.58	8.8	6	2.50	9.8	4	4.55	21.4	5	6.6	30.6	5	8.6	36.6	4	9.4	38.4	4	10.6	66.0	6
No. 252	4.50	.98	13.5	13	2.00	10.9	5	3.95	21.8	5	6.0	32.5	5	8.0	38.7	5	8.8	44.4	5	10.0	47.4	5
No. 298	7-70	0.75	5.3	7	1.70	9.5	6	3.8	24.0	6	4.8	22.4	5	4.8	24.4	5	5.6	26.3	5	-	-	-
No. 299	7-00	1.50	5.2	3	2.40	12.0	5	4.5	24.7	5	5.5	27.7	5	5.5	29.6	5	6.3	32.3	5	-	-	-
No. +101[a]....	0	1.48	13.4	9	1.94	13.4	7	2.93	14.5	5	3.48	18.1	5	6.48	21.8	3	12.5	43.5	4	13.3	40.8	3
No. +105[a]....	0	1.48	13.0	9	1.94	13.5	7	2.93	17.0	6	3.48	16.5	5	6.48	24.2	4	12 5	46.6	4	13.3	46.5	4
No. +102	0	1.48	13.5	9	1.94	14.0	7	2.93	13.8	5	3.48	18.4	5	6.48	22.4	3	12.5	38.4	3	13.3	38.7	3
No. +106	0	1.48	4.0	3	1.94	8.8	4	2.93	9.0	3	3.48	9.6	3	6.48	22.1	3	12.5	30.0	2	13.3	33.3	2
No. 115	0.65	.83	0.6	0	1.30	5.9	5	2.83	9.9	4	4.83	15.3	3	6.85	20.9	3	11.9	31.9	3	12.7	32 6	3
No. 117	0.75	.73	5.7	8	1.20	5.7	5	2.73	7.2	3	4.73	13.3	3	6.75	19.6	3	11.8	25.8	2	12.6	29.2	2
No. 125	1.45	0	0	0	.50	3.0	6	3.47	13.9	4	5.00	12.2	2	11.0	31.2	3	11.8	33.7	3	13.1	33.2	3
No. 127[a]....	1.70	0	-2.8	0	.24	-0.8	0	3.22	18.7	6	4.8	17.9	4	10.8	42.0	4	11.6	39.0	3	12.8	46.7	4
No. 135	2.60	0	3.1	0	2.32	12.4	5	2.88	16.5	6	4.9	20.8	4	9.9	29.0	3	10.7	31.9	3	11.9	32.1	3
No. 138	2.00	.45	2.0	4	2.92	9.7	3	3.48	14.3	4	5.5	16.6	3	10.5	25.8	3	11.3	26.8	2	12.5	25.9	2
No. 139	1.90	.55	1.6	3	3.02	11.2	4	3.58	16.1	4	5.6	16.4	3	10.6	28.1	3	11.4	29.7	3	12.6	30.5	2
No. 148	2.75	2.17	7.1	3	2.73	11.6	3	3.75	9.6	3	8.7	24.3	3	9.8	27.2	3	10.6	27.4	3	11.7	26.7	2
No. 149	2.75	2.17	6.3	3	2.73	13.1	5	3.75	9.9	3	8.7	23.8	3	9.8	24.6	3	10.6	25.2	2	11.7	27.1	2
No. 800	3.90	1.02	6.9	7	1.58	9.4	6	2.60	7.9	3	4.5	16.1	4	6.6	23.7	4	8.6	24.2	3	9.4	27.5	3
No. 151	3.90	1.58	6.6	4	2.50	7.1	3	4.55	13.2	3	7.6	20.8	3	8.6	19.4	2	9.4	20.6	2	10.6	25.1	2

500 Series - 14 1/2 gauge   400 Series - 12 1/2 gauge   300 Series - 11 gauge   200 Series - 9 gauge   100 Series - 6 gauge

[a] Low copper content wires, 0.05 to 0.08 per cent copper. All other wires have about 0.25 per cent copper.

+ Uncoated wires. All other wires had zinc coating.

TABLE VI.—BREAKING LOADS AND ELONGATION OF UNFABRICATED WIRE SPECIMENS.

Wire Lot	Breaking Load, lb	Elongation in 10 in., per cent	Wire Lot	Breaking Load, lb	Elongation in 10 in., per cent
TESTS ON UNCOATED SPECIMENS EXPOSED 12.45 YR AT BRIDGEPORT, CONN.					
No. 400	350	10.9	No. 405	325	8.7
No. 401	300	9.7	No. 406	360	12.4
TESTS ON UNCOATED SPECIMENS EXPOSED 12.48 YR AT PITTSBURGH, PA.					
No. 200	285	6.2	No. 101	1310	2.6
No. 202	660	1.2	No. 102	1290	6.3
No. 205	400	7.3	No. 105	1475	1.3
No. 206	645	3.1	No. 106	1680	1.5
TESTS ON UNCOATED SPECIMENS EXPOSED 13.31 YR AT PITTSBURGH, PA.					
No. 101	1370	2.1	No. 105	1475	1.0
No. 102	1285	6.0	No. 106	1600	1.1
TESTS ON COPPER-COATED SPECIMENS EXPOSED 12.48 YR AT PITTSBURGH, PA.					
No. 021	1665	2.7	No. 022	2300	2.0
TESTS ON LEAD-COATED SPECIMENS EXPOSED 12.48 YR AT PITTSBURGH, PA.					
No. 040	1635	4.4	No. 042	540	2.4
No. 041	920	4.6	No. 043	100	0.3
TESTS ON COPPER AND LEAD-COATED SPECIMENS EXPOSED 14.45 YR AT PITTSBURGH, PA. (Wires Removed in 1951)					
No. 021	1640	—	No. 040	1790	9.5
No. 022	2320	—	No. 041	1070	12.8
TESTS ON CORROSION-RESISTANT SPECIMENS EXPOSED 12.48 YR AT PITTSBURGH, PA.					
No. 001	1250	2.5	No. 003	1310	0.2
No. 004	2340	2.9	No. 008	2725	0.8
No. 005	310	1.4	No. 009	290	0.6
No. 002	1305	2.8	No. 015	1310	0.8
No. 006	2565	4.7			
No. 007	275	1.3			
TESTS ON CORROSION-RESISTANT STEEL SPECIMENS EXPOSED 14.45 YR AT PITTSBURGH, PA. (Wires Removed in 1951)					
No. 001	1270	2.5	No. 006	2670	3.9
No. 002	1315	2.6	No. 007	280	0.8
No. 003	1295	0.2	No. 008	2610	0.4
No. 004	2472	2.9	No. 009	225	0.2
No. 005	295	1.3	No. 015	1305	0.4

Wire Lot	Exposure to 1R, yr	Breaking Load, lb	Elongation in 10 in., per cent	Wire Lot	Exposure to 1R, yr	Breaking Load, lb	Elongation in 10 in., per cent
TESTS ON ZINC-COATED SPECIMENS EXPOSED 12.45 YR AT BRIDGEPORT, CONN.							
No. 510	2.5	295	0.4	No. 410	1.8	440	8.2
No. 511	1.8	160	5.7	No. 411	2.3	480	2.6
No. 512	1.6	180	3.3	No. 412	2.5	640	1.0
No. 520	2.5	210	8.4	No. 421	2.7	375	10.4
No. 521	3.4	180	8.5	No. 422	2.7	410	10.9
No. 522	3.4	320	0.3	No. 423	2.5	330	14.0
No. 530	5.4	220	5.0	No. 282	2.8	1225	9.5
No. 531	4.7	310	0.4	No. 215	1.8	1055	12.1
No. 540	6.2	225	2.7	No. 216	2.8	1440	5.9
No. 541	4.9	255	8.1	No. 217	2.7	970	9.3
No. 542	5.5	340	0.5	No. 115	1.7	2485	5.3
No. 543	5.9	220	3.6	No. 117	2.7	2050	12.5
No. 800	9.5	1365	4.9	No. 801	8.5	1365	4.9

REFERENCE INFORMATION

ON UNFABRICATED WIRE, BARBED WIRE AND FENCE IN THE WIRE INSPECTION REPORT.

The Wire Test Committee responsible for planning and arranging the exposure tests of specimens covered in this report recommends that the following statement be made in all reports relating to the samples of wire and wire products under exposure tests:

> "It is emphasized that the weight of coating ranges shown under each weight group have been arbitrarily chosen and purposely held to close limitations by the Committee and do not necessarily individually represent any manufacturers' commercial product. On the other hand, the entire range of coatings covered by all weight groups as a whole are designed to include a sufficient range of coating to furnish the useful corrosion resistance data desired."

SYSTEM OF NUMBERING SPECIMENS

Zinc coated and uncoated steel specimens (all have 3-digit numbers)..................(*c*)

When the first digit is.......	5	4	3	2	1
the respective gages are:	14.5	12.5	11	9	6

The second and third digits indicate weight of zinc coating as follows:

	oz. per sq.ft.		oz. per sq.ft.
00–04 (LC)*	uncoated	50–59 (CB)	1.6 to 1.8
05–09 (CB)*	uncoated	60–64 (CB)	1.0 to 1.2
10–14 (CB)	0.20 to 0.30	65–69 (CB)	1.3 to 1.5
15–19 (CB)	0.25 to 0.35	70–74 (CB)	1.6 to 1.8
20–24 (CB)	0.35 to 0.45	75–79 (CB)	2.4 to 2.6
25–29 (CB)	0.45 to 0.55	80–84 (CB)	less than 0.50
30–34 (CB)	0.50 to 0.60	85–89 (CB)	1.2 to 1.5
35–39 (CB)	0.60 to 0.70	90–94 (CB)	2.4 to 3.0
40–44 (CB)	0.70 to 0.85	97–99 (CB)	extra heavy
		95–96 (CB)	chromated after coating
45–49 (CB)	0.80 to 1.00 on farm fence and unfabricated wire. 0.85 to 1.00 on barbed wire. 0.80 to 0.95 on wire strand.		

Specimens of Special Materials:
000–015: uncoated corrosion-resistant steel.
020–030: copper-covered steel.
040–050: lead-coated steel.

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* LC = low copper content wire; CB = copper bearing wire

Reference Characters

(*a*) .....Wire lot numbers identify wire from a coil or coils of wire supposedly alike.

Wire Lots of Zinc-Coated or Bare Mild Steel	Usually Appear in the Test as shown by ✓				
	Unfab. + Wire	Int. of + Fence	T and B + of Fence	T and B of Fence	Barbed Wire
100 series	✓ (S)	....	....	....	....
200 series	✓ (S)	✓ (S)	✓ (S)	✓ (300)	....
300 series	✓ (S)	✓ (S)	....	✓ (500)	....
400 series	✓ (S)	✓ (S)	....	....	✓ (400)
(*s*)...... 500 series	✓ (S)	✓ (S)	....	....	....

(S) The specimen numbers of the unfabricated wire or fence are the same as the wire lot numbers.

(300), (400), (500): Specimens of fence numbered in the series 300 have top and bottom wires of the 200 series wire lots; fences numbered in the 500 series have top and bottom wires of the 300 series wire lots. Specimens of barbed wire (numbered in the 400 series) have wire lots of the 400 series but not necessarily of the same identical wire lot numbers.

Par. A. The zinc-coated wire lots table below shows:

– (1) All the individual numbered wire lots and the various utilizations of each wire lot in the test.

(2) The results of weight of coating tests on samples cut from each source (unfabricated wire, fence interiors, fence top and bottom wires, and barbed wires).

(*b*)...... (3) The average coating for each wire lot which is the average of all the figures in the third and following columns in each row.

These average coating figures are the ones entered in other tables of this report. For example, wherever unfabricated wire sample No. 317 or farm fence sample No. 317 are listed, their indicated weights of coating are 0.38 oz. per sq.ft., and not 0.46 and 0.34, respectively.

These wire lot and coating reference data provide an indication of the uniformity of coating on any wire lot.

Par. B. The makeup of fences table below shows:

(1) The lot number of the wire used in the top and bottom line wires of each fence. (The lot number of the wire used in the interior line and stay wires of any fence is the same as the fence number.)

(2) The type of fence, that is, the combination of gages used in its fabrication as explained below:

(*d*)......				
Type of fence	4	3	2	1
Gage of interior line and stay wires	14.5	12.5	11	9
Gage of top and bottom line wires	11	10	9	9

+ ......Abbreviations: Unfab., unfabricated wire; Int., interior line and stay wire; T and B, top and bottom line wires.

Zinc-Coated Wire Lots: Their Coating Weights and Test Specimens in Which They Are Used
(See Par. A and reference characters above for explanations) (Coating weights, oz. per sq.ft.)

Wire Lot (*a*) (Fence)	(*b*) Average Coating	Coating Weight Tests on Samples Cut From Unfab. Wire(*s*)	Coating Weight Tests on Samples Cut From Fence Int.(*s*)	Barbed Wire Coating	Barbed Wire No.
No. 510(*c*)	0.29	0.29	0.29		
No. 511	0.24	0.24	0.23		
No. 512	0.27	0.27	0.27		
No. 520	0.36	0.34	0.38		
No. 521	0.38	0.40	0.36		
No. 522	0.44	0.44	0.44		
No. 530	0.61	0.59	0.62		
No. 531	0.60	0.58	0.61		
No. 532	0.49	....	0.49		
No. 540	0.70	0.72	0.68		
No. 541	0.76	0.80	0.72		
No. 542	0.70	0.69	0.71		
No. 543	0.79	0.83	0.74		
No. 551	1.69	1.69	....		
No. 552	2.08	2.08	....		
No. 410(*c*)	0.30	0.25	0.38	0.27	(410)
No. 411	0.28	0.28	0.28	....	....
No. 412	0.29	0.30	0.30	0.27	(412)
No. 421	0.47	0.49	0.49	0.44	(422)
No. 422	0.35	0.38	0.31	....	....
No. 423	0.31	0.35	0.31	0.28	(420)
No. 430	0.54	0.56	0.52	0.53	(431)
No. 431	0.48	0.49	0.48	0.46	(430)
No. 432	0.57	0.58	0.60	0.53	(432)
No. 433	0.49	....	0.49	....	....
No. 440	0.76	0.72	0.79	....	....
No. 441	0.78	0.77	0.76	0.82	(442)
No. 442	0.72	0.65	0.86	0.64	(441)
No. 443	0.74	0.74	0.78	0.70	(443)
No. 444	0.87	0.87	....	....	....
No. 450	1.50	1.48	....	1.52	(452)
No. 451	1.83	1.71	....	1.95	(451)
No. 495 Cr	Made from unfab. wire 443.				

	Unfab. Wire		Unfab. Wire
No. 115(*c*)	0.25	No. 139	0.64
No. 117	0.27	No. 148	0.90
No. 125	0.41	No. 149	0.85
No. 127	0.54	No. 151	1.86
No. 135	0.74	No. 800(6 ga.)	1.35
No. 138	0.67	No. 801(9 ga.)	1.20

Wire Lot (*a*) (Fence)	(*b*) Average Coating	Coating Weight Tests on Samples Cut From Unfab. Wire(*s*)	Fence Int.(*s*)		T and B of Fence Top	T and B of Fence Bottom	T and B of Fence No.
No. 315(*c*)	0.25	0.25	0.25		0.25	0.26	(512)
No. 316	0.32	0.32	0.31		0.32	0.33	(510)
No. 317	0.38	0.46	0.34		0.37	0.36	(511)
No. 325	0.44	0.50	0.52		0.37	0.39	(520)
No. 326	0.38	0.40	0.35		....	....	....
No. 327	0.47	0.48	0.44		0.48	0.48	(522)
No. 329	0.54	0.56	....		0.45	0.61	(521)
No. 335	0.61	0.61	0.63		0.59	0.60	(531)
No. 336	0.66	0.73	0.60		....	....	....
No. 337	0.57	0.66	0.59		0.50	0.52	(532)
No. 338	0.69	0.67	0.76		0.67	0.65	(530)
No. 345	0.84	0.83	0.97		0.76	0.79	(541)
No. 346	0.88	0.84	0.84		0.96	0.88	(542)
No. 347	0.88	0.96	1.00		0.78	0.79	(543)
No. 348	1.05	1.04	1.07		1.05	1.06	(540)
No. 350	1.75	1.73	1.76		....	....	....
No. 351	1.65	1.65	....		....	....	....
No. 352	1.75	1.82	1.68		....	....	....
No. 397	2.80	2.80	2.79		....	....	....
			Fence(*s*) T-B Top	Fence(*s*) T-B Bottom	T and B of Fence Top	T and B of Fence Bottom	No.
No. 215(*c*)	0.28	0.28	0.25	0.25	0.33	0.29	(315)
No. 216	0.28	0.31	0.30	0.31	0.27	0.29	(316)
No. 217	0.36	0.29	....	....	0.40	0.39	(317)
No. 225	0.45	0.45	0.49	0.44	0.43	0.47	(327)
No. 226	0.45	0.46	....	....	0.45	0.45	(326)
No. 227	0.51	0.59	0.48	0.49	....	....	
No. 228	0.50	0.49	0.48	0.48	0.50	0.53	(325)
No. 235	0.67	0.82	0.60	0.62	0.68	0.64	(337)
No. 236	0.68	0.66	0.68	0.73	0.69	0.65	(338)
No. 237	0.63	0.72	0.67	0.55	0.58	0.61	(335)
No. 238	0.60	0.60	0.59	0.61	0.59	0.61	(336)
No. 239	0.65	0.65	....	....	....	....	....
No. 245	0.81	0.80	0.83	0.78	0.81	0.81	(346)
No. 246	0.95	0.97	0.94	0.98	0.89	0.95	(347)
No. 247	0.96	0.94	0.95	1.00	1.01	0.91	(345)
No. 248	0.81	0.66	0.92	0.68	0.91	0.89	(348)
No. 250	1.76	1.76	....	....	....	....	....
No. 251	1.86	1.68	....	....	1.99	1.91	(352)
No. 252	1.69	1.67	1.85	1.57	....	....	(350)
No. 282	0.37	0.37	....	....	....	....	....
No. 296 Cr	Made from unfab. wire 245.						
No. 298	2.72	2.78	2.71	2.74	2.71	2.67	(397)
No. 299	3.00	3.00	....	....	....	....	....

Makeup of Fences
(See Par. B and reference characters above)

Int. Lot (Fence)	Type (*d*)	T-B Lot	Int. Lot (Fence)	Type (*d*)	T-B Lot
ZINC-COATED FENCES					
No. 510	4	No. 316	No. 410	3	....
No. 511	4	No. 317	No. 411	3	....
No. 512	4	No. 315	No. 412	3	....
No. 520	4	No. 325	No. 421	3	....
No. 521	4	No. 329	No. 422	3	....
No. 522	4	No. 327	No. 423	3	....
No. 530	4	No. 338	No. 430	3	....
No. 531	4	No. 335	No. 431	3	....
No. 532	4	No. 337	No. 432	3	....
No 540	4	No. 348	No. 433	3	....
No. 541	4	No. 345	No. 440	3	....
No. 542	4	No. 346	No. 441	3	....
No. 543	4	No. 347	No. 442	3	....
			No. 443	3	....
No. 315	2	No. 215			
No. 316	2	No. 216	No. 215	1	No. 215
No. 317	2	No. 217	No. 216	1	No. 216
No. 325	2	No. 228	No. 225	1	No. 225
No. 326	2	No. 226	No. 227	1	No. 227
No. 327	2	No. 225	No. 228	1	No. 228
No. 335	2	No. 237	No. 235	1	No. 235
No. 336	2	No. 238	No. 236	1	No. 236
No. 337	2	No. 235	No. 237	1	No. 237
No. 338	2	No. 236	No. 238	1	No. 238
No. 345	2	No. 247	No. 245	1	No. 245
No. 346	2	No. 245	No. 246	1	No. 246
No. 347	2	No. 246	No. 247	1	No. 247
No. 348	2	No. 248	No. 248	1	No. 248
No. 350	2	No. 252	No. 252	1	No. 252
No. 352	2	No. 251	No. 298	1	No. 298
No. 397	2	No. 298			
BARE-STEEL FENCES					
No. 505	4	No. 306	No. 305	2	No. 206
No. 506	4	No. 305	No. 306	2	No. 207
No. 405	3	....	No. 205	1	No. 205
No. 406	3	....	No. 206	1	No. 206
Coppered-covered steel fence No. 020			Int. No. 021	2	No. 022
Lead-coated steel fence No. 040			Int. No. 041	1	No. 041

Uncoated Corrosion-Resistant Steel Fences

No. 001 (T - B Lot No. 001) Gages: Int. 12 ; T - B 12
No. 002 (T - B Lot No. 002) Gages: Int. 12.5; T - B 12.5
No. 003 (T - B Lot No. —) Gages: Int. 15 ; T - B 13

PLATE II
PROCEEDINGS, AM. SOC. TESTING MATS,
VOL 51.
REPORT OF COMMITTEE A-5:
WIRE INSPECTION REPORT

TABLE VI.—*Continued.*

Wire Lot	Exposure to 1R, yr	Breaking Load, lb	Elongation in 10 in., per cent	Wire Lot	Exposure to 1R, yr	Breaking Load, lb	Elongation in 10 in., per cent
ZINC-COATED UNFABRICATED WIRE SPECIMENS. TESTS ON ZINC-COATED SPECIMENS EXPOSED 12.44 YR AT SANDY HOOK, N. J.							
No. 410	1.8	320	3.8	No. 215	3.7	885	7.7
No. 411	1.8	360	0.6	No. 216	2.5	1230	10.3
No. 412	2.5	480	0.3	No. 217	2.8	840	8.5
No. 421	2.8	265	4.7	No. 520	2.7	120	1.5
No. 422	2.9	330	7.3	No. 521	2.9	90	0.6
No. 423	2.7	235	6.9	No. 522	2.9	160	0.3
No. 430	4.4	450	0.4	No. 530	2.9	185	0.3
No. 431	3.7	535	0.5	No. 531	4.7	150	1.4
No. 432	6.5	365	3.8	No. 282	2.8	1045	7.4
TESTS ON ZINC-COATED SPECIMENS EXPOSED 12.48 YR AT BRUNOT ISLAND (PITTSBURGH, PA.).							
No. 345	2.7	225	2.7	No. 298	7.7	1180	1.1
No. 346	2.5	270	0.5	No. 299	7.0	1330	0.6
No. 347	2.6	200	2.2	No. 227	2.5	880	1.6
No. 348	3.2	335	0.8	No. 800	3.9	2025	3.5
No. 350	5.1	300	3.6	No. 801	3.5	935	2.3
No. 351	2.9	410	...	No. 248	2.1	830	0.9
No. 352	3.9	585	1.9	No. 296	2.5	970	1.3
No. 397	7.5	580	0.3	No. 298	7.7	1150	2.1
No. 215	0.6	495	7.1	No. 299	7.0	1295	0.8
No. 216	1.3	805	1.6	No. 115	0.6	1850	2.7
No. 217	1.0	625	5.2	No. 117	0.7	1645	8.6
No. 225	1.9	890	1.3	No. 125	1.5	1265	10.7
No. 226	1.9	680	9.6	No. 127	1.7	1475	2.1
No. 227	2.5	875	1.9	No. 135	2.6	1890	2.3
No. 228	1.6	660	10.0	No. 138	2.0	1455	12.1
No. 235	2.6	730	5.5	No. 139	1.9	1380	8.6
No. 236	2.0	755	1.2	No. 148	2.7	1855	3.2
No. 237	1.8	870	1.3	No. 149	2.7	1440	11.2
No. 238	2.5	895	1.6	No. 151	3.9	2500	1.1
No. 239	2.6	680	12.3				
No. 245	2.4	815	1.2				
No. 246	2.9	615	8.1				
No. 247	2.9	985	1.7				
TESTS ON ZINC-COATED SPECIMENS EXPOSED 13.31 YR AT BRUNOT ISLAND (PITTSBURGH, PA.).							
No. 350	5.1	210	3.9	No. 251	3.9	1195	0.5
No. 351	2.9	355	3.1	No. 252	4.5	640	5.9
No. 352	3.9	490	0.1	No. 298	7.7	1120	1.2
No. 397	7.5	595	0.4	No. 299	7.0	1245	0.5
No. 225	1.9	800	1.4	No. 115	0.6	1830	3.2
No. 226	1.9	615	8.8	No. 117	0.7	1570	8.5
No. 227	2.5	765	1.8	No. 125	1.4	1220	13.6
No. 228	1.6	565	10.7	No. 127	1.7	1550	2.1
No. 235	2.6	870	2.7	No. 135	2.6	1805	2.3
No. 236	2.0	715	0.9	No. 138	2.0	1435	11.1
No. 237	1.8	750	1.7	No. 139	1.9	1350	9.3
No. 238	2.5	820	1.8	No. 148	2.7	1850	2.7
No. 239	2.6	610	11.7	No. 149	2.7	1430	11.2
No. 248	2.1	720	0.5	No. 151	3.9	2460	0.7
No. 296	2.5	875	1.3	No. 800	3.9	1935	3.5
No. 250	3.9	825	6.1	No. 801	3.5	865	0.9
TESTS ON ZINC-COATED SPECIMENS EXPOSED 14.45 YR AT PITTSBURGH, PA. (Wires Removed in 1951)							
No. 125	1.4	1230	9.5	No. 149	2.8	1380	12.0
No. 127	1.7	1355	1.3	No. 151	3.9	2320	0.6
No. 135	2.6	1805	2.0	No. 250	3.9	785	5.9
No. 138	2.0	1455	12.1	No. 251	3.9	1055	0.4
No. 139	1.9	1335	9.3	No. 252	4.5	605	5.6
No. 148	1.8	1870	3.2				

Findings from Weight-Loss Specimens:

At the start of the test, zinc-coated unfabricated wire specimens were exposed at Pittsburgh, Sandy Hook and State College to determine the rate of loss of coating. These tests have now been completed and summarized in the 1947 report.[2] The over-all average annual loss of coating in ounces per square foot of surface at Pittsburgh is 0.369; at Sandy Hook it is 0.117; and at State College it is 0.060.

Respectfully submitted on behalf of the Wire Test Inspection Committee,

A. P. JAHN,
Chairman.

REPORT OF COMMITTEE A-7

ON

MALLEABLE IRON CASTINGS*

Committee A-7 on Malleable Iron Castings held one meeting during the current year. This was held at the time of the Annual Meeting on June 26, 1950.

A meeting of the special Subcommittee on Malleable Iron Flanges, Pipe Fittings and Valve Parts was held in Cincinnati, Ohio, at the time of the Spring Committee Week Meeting on March 5, 1951.

At the present time the committee consists of 55 members, one of whom is classified as non-voting; of the 54 voting members, 27 are classified as producers, 14 as consumers, and 13 as general interest members.

Committee A-7 regrets to record the death of Carl F. Lauenstein, former vice chairman of this committee. Through the tragic death of our associate, the committee loses the advice and council of an able metallurgist, an excellent committeeman and an extremely good friend.

Committee A-7 is still continuing its general interest in the project of working out some arrangement with the National Bureau of Standards by which a White Iron Sample will be added to the Bureau's list of standard samples. Some progress has been made during the current year, but the pressure of present-day conditions on the Bureau is introducing some new causes for interference with the completion of this project.

SUBCOMMITTEE ON MALLEABLE IRON FLANGES, PIPE FITTINGS AND VALVE PARTS

As a result of further study of the Tentative Specification for Malleable Iron Flanges, Pipe Fittings and Valve Parts (A 277 – 44 T), it has been decided to substitute for that specification a new tentative with the title "Malleable Iron Flanges, Pipe Fittings and Valve Parts for Railroad, Marine and Other Heavy Duty Service," with the understanding that this specification will be issued under a new designation number.

This proposed tentative specification, which will be under the jurisdiction of Subcommittee V on Malleable Iron Flanges, Pipe Fittings and Valve Parts for Railroad, Marine and Other Heavy Duty Service, has been submitted to letter ballot of the committee with returns to date showing 30 affirmative, 6 not voting and 0 negative votes. Of those voting in the affirmative, 15 are listed as producers, 7 as consumers and 8 in the general interest classification.

It is expected that these specifications will be submitted to the Administrative Committee on Standards soon after this Annual Meeting.

This report has been submitted to letter ballot of the committee, which consists of 55 members; 40 members returning their ballots of whom 38 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

W. A. KENNEDY,
Chairman.

J. H. LANSING,
Secretary.

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* Presented at the Fifty-fourth Annual Meeting of the Society, June 18–22, 1951.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee A-7 presented to the Society through the Administrative Committee on Standards the following recommendations:

Tentative Specifications for:

Malleable Iron Flanges, Pipe Fittings, and Valve Parts for Railroad, Marine and Other Heavy Duty Service (A 338 - 51 T).

Withdrawal of Tentative Specifications for:

Malleable Iron Flanges, Pipe Fittings, and Valve Parts (A 277 - 44 T).

These recommendations were accepted by the Standards Committee on September 12, 1951, and the new specifications appear in the 1951 Supplement to Book of ASTM Standards, Part 1.

REPORT OF COMMITTEE A-10

ON

IRON-CHROMIUM, IRON-CHROMIUM-NICKEL AND RELATED ALLOYS*

Committee A-10 on Iron-Chromium, Iron-Chromium-Nickel, and Related Alloys held meetings at Atlantic City, N. J., on June 29, 1950, during the Annual Meeting of the Society, and on March 6, 1951, at Cincinnati, Ohio.

The committee expresses its appreciation to H. D. Newell, former secretary of A-10, for outstanding services rendered from 1930 to 1950 as secretary of the committee.

The present committee consists of 91 members, of whom 41 are classified as producers, 36 as consumers, and 14 as general interest members.

Committee A-10 has approved by letter ballot the following Emergency Alternate provision for submission to the Administrative Committee on Standards:

> In view of the limited availability of columbium (with the heretofore usual small percentage of tantalum) the stabilizing addition to type— shall consist of columbium plus tantalum. The ratio of columbium to tantalum (in relation to the carbon content of the steel) and the maximum of each element, or of one of them, or of the two combined, shall be determined in accordance with the requirements of the end use and shall be as mutually agreed upon by producer and purchaser.

This provision affects the following specifications:

Standard Specifications for:

Corrosion-Resisting Chromium-Nickel Steel Plate, Sheet, and Strip (A 167 – 44),
Corrosion-Resisting Chromium and Chromium-Nickel Steel Plate, Sheet, and Strip for Fusion-Welded Unfired Pressure Vessels (A 240 – 49),
Seamless and Welded Austenitic Stainless Steel Tubing for General Service (A 269 – 47), and
Seamless Austenitic Chromium-Nickel Steel Still Tubes for Refinery Service (A 271 – 47).

Tentative Specifications for:

Hot-Rolled and Cold-Finished Corrosion-Resisting Steel Bars (A 276 – 49 T),
Corrosion-Resistant Iron-Chromium and Iron-Chromium-Nickel Alloy Castings for General Application (A 296 – 49 T),
Seamless and Welded Austenitic Stainless Steel Pipe (A 312 – 48 T), and
Corrosion-Resisting Steel Billets and Bars for Reforging (A 314 – 47 T).

It has also under consideration an Emergency Alternate provision to Specification A 312 to read as follows:

> The phosphorus content of all grades covered in this specification is increased to 0.045 per cent check analysis for the duration of the present emergency.

ADOPTION OF TENTATIVE AS STANDARD

The committee reports a favorable letter ballot confirming the action taken by Committee B-4 recommending that the Tentative Specification for Chromium-Nickel-Iron Alloy Castings (25-12 Class) for High-Temperature Service (B 190 – 45 T)[1] be adopted as standard without revision. This recommendation was approved by the Society in 1950 and the specification appears in the 1950 Supplement to Book of ASTM Standards, Part 2, under the designation B 190-50. This specification was originally prepared in cooperation with Committee B-4 on Electrical Heating, Resistance, and Related Alloys, and is under the joint jurisdiction of the two committees.

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* Presented at the Fifty-fourth Annual Meeting of the Society, June 18–22, 1951.

[1] 1949 Book of ASTM Standards, Part 1.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Classification of Data (F. P. Peters, chairman).—F. P. Peters has resigned as chairman of Subcommittee I; T. C. DuMond was nominated to replace F. P. Peters as chairman of the subcommittee.

Subcommittee IV on Methods of Corrosion Testing (F. L. LaQue, chairman) has adopted by letter ballot revisions of the Tentative Recommended Practice for Boiling Nitric Acid Test for Corrosion-Resisting Steels (A 262 - 50 T). The revisions involve: (*a*) the addition of a footnote suggesting metallographic examination of specimens that have suffered a high weight loss in the test to establish whether intergranular attack actually has occurred; (*b*) an amendment to permit use of multiple sample testing apparatus,[2] and (*c*) permission of the use of a nylon brush as an alternate to rubber in cleaning specimens after exposure to the acid.

The revision indicated in item (*a*) above was proposed to the Administrative Committee on Standards for adoption in 1950. This committee referred it back to Subcommittee IV for a reconciliation of a negative vote. This having been done, Subcommittee IV will seek approval of Committee A-10 at the 1951 Annual Meeting for submission of these proposed revisions to the Society for approval by the Administrative Committee on Standards.

A special sub-group, under the chairmanship of E. G. Holmberg, is preparing a recommended practice for cleaning of stainless steel surfaces, particularly in connection with fabricated equipment.

Further attention has been given to the proposed atmospheric corrosion test program, but current difficulties in the procurement of specimen material has delayed inauguration of these tests.

Subcommittee IV does not recommend revision of Tentative Method of Total Immersion Corrosion Test of Stainless Steels (A 279 - 44 T). This method should be continued as tentative until its possible combination with Tentative Method of Total Immersion Corrosion Test of Non-Ferrous Metals (B 185 - 43 T) has been fully explored.

Subcommittee V on Mechanical Testing (R. H. Heyer, chairman) cooperated with Subcommittee IX in an investigation of alternate specimen shapes for tension testing of stainless steel plates. The joint task group reported at the March, 1951, meeting, recommending that Specifications A 167, A 176, and A 240 be reviewed and revised to provide adequate illustrations and descriptive matter on tension testing of plates, and to include provisions for a modified specimen shape based on the results of the cooperative investigation.

A test program is under way for determining the effect of rates of strain between 0.05 and 1.00 in. per in. per min on the tensile properties of Type 301 strip, as outlined at the June, 1950, meeting of the subcommittee.

Subcommittee VI on Metallography (Russell Franks, chairman).—The most important activity of Subcommittee VI during the past year was the job of correlating the data obtained on 18-8 steels (with and without molybdenum) in an effort to identify the sigma phase after heating these steels to elevated temperatures.

The various etching procedures that have been employed on different samples of the steels in the attempt to identify the sigma phase have been studied and progress has been made. On the basis of this work, it may be stated that certain of the procedures will definitely identify the sigma phase, provided the composition of the steel and the heat treatments to which it has been subjected are known.

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[2] Described by W. B. DeLong in *STP No. 93* (1949).

This result has been accomplished in conjunction with X-ray diffraction tests which give definite proof of the presence or absence of the sigma phase.

Subcommittee VIII on Specifications for Wrought Products (J. K. Findley, chairman) has under consideration a specification for stainless steel strand wire. It is expected that action on this specification will be taken at the June, 1951, meeting of the subcommittee.

Subcommittee IX on Specifications for Flat Products (H. A. Grove, chairman).—A special sub-group has collected data on the effect of the present 60:40 ratio of Columbium-tantalum ferro-alloy to replace ferro-columbium in type 347 steel, with the thought of permitting its use in all specifications covering this analysis in both cast and wrought products. One report has been submitted to the chairman of A-10 for distribution to the members of the committee. The data in this report indicate that the Cb-Ta composition could be substituted for the regular columbium content in type 347 without affecting the mechanical or physical properties of this grade.

The work of the special subcommittee on the shape and size of tension test specimen for plates over $\frac{3}{4}$ in. thick has been completed. A report covering this work was presented and approved at the March, 1951, meeting of the subcommittee in Cincinnati and shows that a full-size plate specimen can be used for plate $\frac{3}{4}$ in. and over in thickness, in place of the $\frac{1}{2}$ in. round specimen now required. The use of the full section specimen should be optional and will affect Specifications A 167, A 176, and A 240. Revision of these specifications has been approved by letter ballot of the subcommittee.

Subcommittee IX is considering revision of the chemical requirements in Specification A 167 – 44 in order to have them conform to present practice as expressed in the AISI ranges. The most critical change in Specification A 167 – 44 concerns the analysis of Grade 6, type 347. This is to permit the use of columbium and tantalum together as a stabilizing addition as well as the present columbium alloy. It was proposed at the June, 1950, meeting that Footnote *b* be changed to read as follows: "Grade 6 shall have a minimum columbium or combined columbium-tantalum content of 10 times the carbon content but not more than 1.35 per cent max." The content of tantalum, if determined, shall not be greater than 0.4 times the sum of the columbium plus tantalum content. Consideration is being given to the addition of a paragraph under Check Analyses to read as follows: "When separate analysis for columbium and tantalum is required then the method of analysis shall be agreed upon by purchaser and vendor."

Footnote *b* under Table I in Specification A 240 – 49 is being revised as above to permit the use of Cb-Ta combined in Grade C of this specification.

The Emergency Alternate Recommendation cited in the beginning of this report is, of course, in addition to and will supersede these permanent changes.

Subcommittee X on Specification for Castings (J. J. Kanter, chairman).—At the March, 1951, meeting in Cincinnati, this subcommittee considered a proposal regarding columbium-tantalum stabilization of stainless steel. It was decided that as regards the casting specifications, no actions or recommendations should be taken until a policy for handling of such provisions has been finally adopted in Committee A-10.

A task group under the chairmanship of W. F. Hodges has been engaged in the formulation of specifications for centrifugally-cast iron-alloy tubing for heat- and corrosion-resisting service. A revised draft of the proposed specifications will be discussed at the June, 1951, meeting of Subcommittee X.

Subcommittee XI on Specifications for Tubular Products (J. J. B. Rutherford, chairman).—The activities of this subcommittee during the past year have involved two different fields: revision of A 312 - 48 T, Tentative Specification for Seamless and Welded Austenitic Stainless Steel Pipe, and review of the situation regarding strategic materials.

Specification A 312 was revised to obtain a definition of the welding process, and thereby eliminate filler metal, in accordance with the requirements of the Boiler Code Committee of ASME. Supplementary tests were added to embody the requirements of pipe for high temperature, high pressure service. A proposal by the Valve and Fittings Industry Committee, to include a new schedule of wall thickness known as Schedule 5s, was considered and accepted on letter ballot of the subcommittee. This makes available an extra light wall pipe, suitable for welding fabrication but not for threading. Two new grades, TP-309 and TP-310, 25Cr - 12Ni and 25Cr - 20Ni, respectively, will be added to this specification.

After consideration of the availability of such elements as manganese, nickel, columbium etc., no specific actions were taken since these problems lie in the realm of possible government control. A request was made of the Society to arrange for a temporary increase of phosphorus content in these grades, to 0.045 per cent check analysis. The phosphorus content of tubular products is lower than in other commodities and this entails a procurement hardship.

The committee was asked to consider the inclusion and definition of a heat treatment clause in Specification A 312. A task group has taken this under consideration. The committee was also asked to promulgate a specification for stainless steel pipe made by fusion welding processes, with filler metal, up to 24-in. size. This matter will be up for preliminary discussion at the June, 1951, meeting.

This report has been submitted to letter ballot vote of the committee, which consists of 96 members; 60 members returned their ballots, of whom 56 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

JEROME STRAUSS,
Chairman.

M. A. CORDOVI,
Secretary.

(See Editorial Note, p. 132)

EDITORIAL NOTE

Subsequent to the Annual Meeting, the following Emergency Alternate Provisions, submitted by Committee A-10, were accepted by the Administrative Committee on Standards on the date indicated:

Designation	Emergency Alternate Provisions In	Date Issued
	Standard Specifications for:	
EA - A 167	Corrosion-Resisting Chromium-Nickel Steel Plate, Sheet, and Strip (A 167 - 44)	Aug. 21, 1951
EA - A 240	Corrosion-Resisting Chromium and Chromium-Nickel Steel Plate, Sheet, and Strip for Fusion-Welded Unfired Pressure Vessels (A 240 - 49)	Aug. 21, 1951
*EA - A 268	Seamless and Welded Ferritic Stainless Steel Tubing for General Service (A 268 - 47)	July 10, 1951
*EA - A 269	Seamless and Welded Austenitic Stainless Steel Tubing for General Service (A 269 - 47)	July 10, 1951 and Oct. 18, 1951
*EA - A 270	Seamless and Welded Austenitic Stainless Steel Sanitary Tubing (A 270 - 50)	July 10, 1951
*EA - A 271	Seamless Austenitic Chromium-Nickel Steel Still Tubes for Refinery Service (A 271 - 47)	July 10, 1951 and Oct. 18, 1951
	Tentative Specifications for:	
EA - A 276	Hot-Rolled and Cold-Finished Corrosion-Resisting Steel Bars (A 276 - 49 T)	Aug. 21, 1951
EA - A 296	Corrosion-Resistant Iron-Chromium and Iron-Chromium-Nickel Alloy Castings for General Application (A 296 - 49 T)	Aug. 21, 1951
EA - A 312	Seamless and Welded Austenitic Stainless Steel Pipe (A 312 - 51 T)	July 10, 1951 and Oct. 18, 1951
EA - A 314	Corrosion-Resisting Steel Billets and Bars for Reforging (A 314 - 47 T)	Aug. 21, 1951

* Jointly with Committee A-1.

These emergency provisions have been published in the ASTM BULLETIN and have also been issued in the form of pink stickers for attachment to the standards to which they apply.

On October 29, 1951, the Administrative Committee on Standards accepted the following recommendations which Committee A-10 submitted jointly with Committee A-1 on Steel:

Revision of Tentative Specifications for:
Seamless and Welded Austenitic Steel Pipe (A 312 - 51 T).

Tentative Revision of Standard Specifications for:
Seamless Austenitic Chromium-Nickel Steel Still Tubes for Refinery Service (A 271 - 47).

REPORT OF COMMITTEE B-1

ON

WIRES FOR ELECTRICAL CONDUCTORS*

Committee B-1 on Wires for Electrical Conductors held three meetings during the year: one at the Society's headquarters in Philadelphia, Pa. on October 11, 1950; one in New York, N. Y., on January 18, 1951; and one in Washington, D. C., on April 20, 1951. Meetings of Subcommittees II, IV, V, VI, and VII also were held during the year.

At the start of this committee year, the assumption of the committee chairmanship by H. H. Stout, Jr., left a vacancy in the chairmanship of Subcommittee IV, which subsequently was filled by B. J. Sirois. Later in the year, due to a change in affiliation, Mr. Stout found it necessary to submit his resignation as committee chairman. Action by the Advisory Committee resulted in the unanimous election of D. Halloran as successor to the chairmanship.

As a result of a long investigation, the committee concluded, and recommended to the Society that a separate compilation of standards should be issued, including all standards under the jurisdiction of Committee B-1 together with a number of related standards and methods. The committee is gratified that this new compilation was published during the year under the title "Standards on Metallic Electrical Conductors."

During the year, organization of a new ASA Sectional Committee, C 7 on Bare Electrical Conductors, was completed under sponsorship of ASTM and action instituted to have a number of ASTM specifications approved as American Standards. Committee B-1 is proud to have taken an active part in this undertaking.

On request from the Cable Engineering Section, Committee on Power Distribution, Association of Edison Illuminating Companies, the committee has been endeavoring to prepare a proposed specification for segmental-type conductors. After almost three years of continued endeavor, it appears that the great variations in commercial production of this product make it impossible to find a suitable basis for standardizing at this time. The Task Group has prepared a summary report of its work, which is appended to this report.

Because of some question as to the accuracy of the cross-sectional areas of shaped trolley wire as shown in Specifications B 9, B 47, and B 116, another Task Group has been engaged for some time in the mathematical recomputation of these values. This work was completed this year and indicated that the values as published are within acceptable accuracy limits. For future reference, the data are appended to this report.

The committee now consists of 75 members, of whom 36 are classified as producers, 28 as consumers, and 11 as general interest members.

NEW TENTATIVE

Early in World War II, Committee B-1 had drafted a proposed specification for hard-drawn and medium-hard-drawn tinned copper wire. Due to materials limitations, the proposed specification was not recommended to the Society but was included with the 1942 Annual Report of the committee.[1]

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* Presented at the Fifty-fourth Annual Meeting of the Society, June 18-22, 1951.

In the past few years there has been a growing consumer demand for this product. Hence the committee has reconsidered this proposal and included certain changes in accordance with current practice. The committee, therefore, recommends for publication as tentative the proposed Specifications for Tinned Hard-Drawn and Medium-Hard-Drawn Copper Wire for Electrical Purposes as appended hereto.[2]

REVISIONS OF TENTATIVES

The committee recommends that the following three specifications be revised as indicated and continued as tentative:

Tentative Specifications for Rope-Lay-Stranded Copper Conductors Having Bunch-Stranded Members for Electrical Conductors (**B 172 - 50 T**).[3]—Revise as follows:

Section 3 (*b*).—Delete reference to ASTM specifications B 174.

Section 5.—Delete the footnote reference 4 after the boldface heading "Lay" and replace with the words "Note 3." Delete the footnote.

In Paragraph (*b*) revise the first sentence to read: "The length of lay of the outer layer of the rope-lay-stranded conductor shall be not less than 8 nor more than 16 times the outside diameter of the completed conductor."

Reletter Paragraph (*c*), as (*d*), and insert a new Paragraph (*c*) to read as follows:

> (*c*) The length of lay of the bunch-stranded and rope-stranded members shall be not more than 30 times the outside diameter of the member. The direction of lay shall be at the option of the manufacturer unless specifically agreed upon.

Section 6.—Revise Paragraph (*a*) to read: "The area of cross-section, and the number and diameter of wires for a variety of strand constructions in general use are shown in Table I (Note 4)."

In Paragraph (*b*) change the word "prescribed" to read "shown."

Section 7.—Revise to read as follows:

> 7. (*a*) Tests for the electrical properties of wires composing conductors made from soft or annealed copper wire, bare or coated, shall be made before stranding.
>
> (*b*) Tests for the physical properties of soft or annealed copper wire, bare or coated, may be made upon the wires before stranding or upon wires removed from the completed stranded conductors, but need not be made upon both. Care shall be taken to avoid mechanical injury and stretching when removing wires from the conductor for the purpose of testing.
>
> (*c*) The physical properties of wire when tested before stranding shall conform to the applicable requirements of Section 3 (*b*).
>
> (*d*) The physical properties of wires removed from the completed stranded conductor shall be permitted to vary from the applicable requirements of Section 3 (*b*) by the following amounts (Note 5):
>
> (*1*) *Average of Results Obtained on All Wires Tested*.—The maximum tensile strength permitted shall be 5 per cent greater than the maximum prescribed for the wires before stranding and the minimum elongation required shall be reduced in numerical value 5 (for example, from 30 per cent to 25 per cent) from the numerical requirements for the wire before stranding.
>
> (*2*) *Results Obtained on Individual Wires*.—The tensile strength of individual wires shall not exceed the maximum prescribed before stranding by more than 15 per cent, and the elongation of individual wires shall be reduced in numerical value 15 from the minimum requirements before stranding (that is, 10 in addition to the 5 allowed in Item (*1*)), but in no case shall the elongation of any individual wire be less than 5 per cent.
>
> (*e*) In the event that the requirements prescribed in Item (*2*) of Paragraph (*d*) are met, but those prescribed in Item (*1*) are not met, a retest shall be permitted wherein all wires of a conductor of 100 wires or less, or 100 wires selected at random throughout a conductor of more than 100 wires shall be tested for the purpose of final determination of conformance to Paragraph (*d*).

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 42, p. 160 (1942).

[2] The new tentative was accepted by the Society and appears in the 1951 Supplement to Book of ASTM Standards, Part 2.

[3] 1949 Book of ASTM Standards, Part 2.

(*f*) By agreement between the purchaser or his representative, and the manufacturer, the tensile strength of the completed stranded conductor as a unit may be determined in lieu of, but not in addition to, physical tests on individual wires before or after stranding. The breaking strength of conductors so tested shall be not greater than 5 per cent higher than the total of the specified maximum breaking strengths of the component wires as set forth in Section 3 (*b*). The maximum breaking strength of the component wires shall be calculated, using nominal specified diameters and maximum specified tensile strengths. The free length between grips of test specimens shall be not less than 10 in. and should be greater than 10 in. for the larger sizes of conductors. Care shall be taken to insure that the wires in the conductor are evenly gripped during the test (Note 6).

(*g*) If a tinning, lead-coating, or lead-alloy-coating test is required, it shall be made on the wires prior to stranding.

Explanatory Notes.—Renumber the present Notes 5 and 6 as 7 and 8, respectively, and correct the references thereto in Sections 8 and 9. Insert two new explanatory notes, as follows:

NOTE 5.—Wires unlaid from conductors manifestly will have different physical and electrical properties from those of the wire when prepared for cabling, on account of the deformation brought about by laying and again straightening for test.

NOTE 6.—To test stranded conductors for tensile strength successfully as a unit requires an adequate means of gripping the ends of the test specimen. Various means are available, such as a long tube or socket into which the conductor may be soldered, or in which, after insertion, the conductor may be swaged or pressed without serious distortion. Ordinary jaws or clamping devices usually are not suitable. The conductor-testing facilities of many commercial laboratories are limited to a breaking strength of 30,000 lb or less. Consequently, it may not be feasible to test the very large-sized conductors as a unit. Where such is imperative, special arrangements for the testing shall be agreed upon between the manufacturer and the purchaser.

Tentative Specifications for Rope-Lay-Stranded Copper Conductors Having Concentric Stranded Members for Electrical Conductors (**B 173 - 50 T**).[3]—Revise as follows:

Section 5.—Delete the footnote reference 4 after the word "Lay" and replace with the words "Note 3." Delete the footnote.

In Paragraph (*b*) make the same change indicated above for Section 5 (*b*) of Specification B 172.

Section 6.—Revise to read: "The area of cross-section, and the number and diameter of wires for a variety of strand constructions in general use are shown in Table I."

Section 7.—Revise as indicated for Section 7 in Specifications B 172, with the exception of referring to Note 4 in Paragraph (*d*), and to Note 5 in Paragraph (*f*).

Explanatory Notes.—Renumber the present notes 4, 5, and 6 as 6, 7, and 8, respectively, and correct the references thereto in Sections 8 and 9 and in the title of Table I. Insert two new explanatory Notes, 4 and 5, to read the same as indicated above for the new Notes 5 and 6 of Specification B 172.

Tentative Specifications for Bunch-Stranded Copper Conductors for Electrical Conductors (**B 174 - 50 T**).[3]—Revise as follows:

Section 6 (*a*).—Revise to read "The area of cross-section, and the number and diameter of wires for a variety of strand constructions in general use are shown in Table III."

Section 7.—Revise as indicated for Section 7 in Specifications B 172, with the exception of referring to Note 3 in Paragraph (*d*), and to Note 4 in Paragraph (*f*).

Explanatory Notes.—Renumber the present Note 3 to read Note 5 and correct the reference thereto in Section 8. Insert two new explanatory Notes 3 and 4, to read the same as indicated above for new Notes 5 and 6 of Specification B 172.

REVISION OF STANDARD AND REVERSION TO TENTATIVE

The committee recommends revision of the Standard Specifications for Hard-

Drawn Copper Wire (B 1 – 49),[3] as appended hereto,[4] and reversion to tentative status as discussed below.

In 1947 a Task Group was formed for the purpose of extracting and unifying the sections in existing specifications on sampling procedure. The initial effort of the group consisted of attempting to write a comprehensive specification for Acceptance Inspection or Sampling, based on a statistical approach, which could be applied to all of the specifications under the jurisdiction of Committee B-1.

Due to the variation in tensile strength and elongation characteristics of the various tempers of copper, it was necessary to have different limiting values for the different tempers, and the only way to obtain these values was to collect data from the various producers.

The Task Group unanimously agreed that it would be desirable to obtain test values on hard-drawn copper first and then to proceed to the other products when a decision had been reached on the first. When the magnitude of the work was realized, the members of the group agreed that, instead of recommending one specification to cover the inspection procedure for all of the specifications, it would be much more practicable to develop a standard sampling procedure and revise the existing B-1 specifications to incorporate this standard procedure.

A review of the general problems associated with an acceptance-inspection procedure applicable to hard-drawn copper wire brought out the following fundamental comments:

1. The hard-drawn copper wire supplied by American industry as a whole during several decades has been found generally satisfactory by consumers.

2. The requirements stated in Specifications B 1, having been used successfully for a great many years for design purposes, should not be altered.

3. An acceptance-inspection procedure should afford a consumer reasonable assurance of satisfactory lot quality without incurring an unreasonable cost of inspection. In this connection, industry's record in delivering satisfactory wire over the years should form a basis for assuming a continuingly satisfactory product.

4. The acceptance-inspection procedure should be a part of Specifications B 1.

These concepts rather definitely define the acceptance procedure which would be applicable. A statistical approach to requirements such as tensile strength was indicated in order to obtain the minimum possible sample size. Before requirements could be expressed on a statistical basis, the properties of the product supplied by industry had to be evaluated in a manner never before attempted.

It is evident from the Task Group's study that the requirements formerly stated as minima do not actually represent minimum test results observed on the product as supplied, but may be considered nominal values for design purposes.

In the opinion of the Task Group, the use of the sampling procedures specified in the attached draft will insure:

1. Essentially continuous conformance of successive lots.

2. That the true average tensile strength and elongation of the product, a succession of lots from which continually meets the acceptance criteria, will exceed the minimum values of tensile strength and elongation given in Table I of Specifications B 1 – 49.

3. That such wire is the equivalent of that supplied and found acceptable for many years.

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[4] The revised specifications were accepted by the Society and appear in the 1951 Supplement to Book of ASTM Standards, Part 2.

REVISIONS OF STANDARDS
IMMEDIATE ADOPTION

The committee recommends, for immediate adoption, revisions in the following three standards and accordingly asks for a nine-tenths afirmative vote at the Annual Meeting in order that these recommendations may be referred to letter ballot of the Society:

Standard Specifications for Tinned Soft or Annealed Copper Wire for Electrical Purposes (**B 33 – 50**).[5]—Revise as follows:

Section 8 (b).—Revise the first sentence to read: "A concentrated solution shall be made by dissolving sodium sulfide cp crystals in distilled water until the solution is saturated at about 21 C (70 F) and adding sufficient flowers of sulfur (in excess of 250 g per liter of solution) to provide complete saturation, as shown by the presence in the solution of an excess of sulfur after the solution has been allowed to stand for at least 24 hr."

Note 7.—Revise the second sentence to read: "A solution which is not saturated with sulfur or which has been made from decomposed sodium sulfide crystals may give a false indication of failure."

Standard Specifications for Lead-Coated and Lead-Alloy-Coated Soft Copper Wire for Electrical Purposes (**B 189 – 50**):[5]

Section 8 (c).—Revise the first sentence to read as indicated above for Section 8 (*b*) of Specification B 33 and add the words "(Note 4)" to the end of the paragraph.

Explanatory Notes.—Renumber the present Note 4 to read Note 5 and correct the reference thereto in Section 11. Insert a new explanatory note, as follows:

NOTE 4.—It is important that the polysulfide solution be of proper composition and strength at the time of test. A solution which is not saturated with sulfur or which has been made from decomposed sodium sulfide crystals may give a false indication of failure. Therefore, the requirement that the solution be tested by observing its blackening effect on a bright copper wire is significant. Significant also is the requirement that the solution be saturated with sulfur by allowing the solution to stand at least 24 hr after preparation. Attention is called also to the necessity for the use of sodium sulfide which has not deteriorated through exposure to air; and if exposure has occurred, the crystals should be tested for purity. The "Standard Reagents Tests" of the American Chemical Society are useful in this connection.

Standard Specifications for Soft Rectangular and Square Bare Copper Wire for Electrical Conductors (**B 48 – 49**).[3]—Revise as follows:

Table II.—Revise to read as shown in the accompanying Table I.

Note 7.—Revise to read as follows:

NOTE 7.—Table II (accompanying Table I) gives data on the cross-sectional area of square wire in sizes 0.0508 to 0.4600 in., inclusive, allowance having been made for reduction of the theoretical area of a perfect square wire due to the rounding of its four corners as shown in Table I of these specifications. These areas are for the nominal dimensions shown in column 1 of Table II (accompanying Table I) and do not take into account the variations in the dimensions of an actual wire as permitted by the tolerances given in these specifications. It has been demonstrated that there is no practical need for expressing working net areas to the same large number of significant figures given, as for instance, in columns 2 or 3. It is felt that the significance of these working net area values should not extend beyond the significance of the values in column 1 and it is for this reason that the working net area values have been rounded off as shown in column 7.—Other information (circular mil area, weight and resistance) often found necessary, can be derived from the data given in Table II (accompanying Table I) as follows:

Nominal Area:
Circular mils = 1.2732 × column 6

Nominal Weight:
Lb per 1000 ft = 0.0038540 × column 6
(Based on copper density 0.32117 lb per cu in.)

Nominal D-C Resistance at 20 C:
Ohms per 1000 ft = 8145.4 ÷ column 6
(Based on a resistivity of 10.371 ohms (mil, ft.)

As in the case of working net areas in sq mils, the values obtained from the use of the foregoing

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[5] 1950 Supplement to 1949 Book of ASTM Standards Part 2.

TABLE I.—AREAS OF SQUARE COPPER WIRE.

Nominal Size, in.	Calculated Area of Perfect Square		Nominal Corner Radius, in.	Calculated Departure,[a] sq mils	Calculated Net Area, sq mils	Net Area Working Value, sq mils
	sq in.	sq mils				
1	2	3	4	5	6	7
0.0508	0.00258064	2580.64	0.012	123.61	2457.03	0.246×10^4
0.0571	0.00326041	3260.41	0.012	123.61	3136.80	0.314×10^4
0.0641	0.00410881	4108.81	0.012	123.61	3985.20	0.399×10^4
0.0720	0.00518400	5184.00	0.012	123.61	5060.39	0.506×10^4
0.0808	0.00652864	6528.64	1/64	209.57	6319.07	0.632×10^4
0.0907	0.00822649	8226.49	1/64	209.57	8016.92	0.802×10^4
0.1019	0.01038361	10383.61	1/64	209.57	10174.04	1.017×10^4
0.1144	0.01308736	13087.36	1/64	209.57	12877.79	1.288×10^4
0.1285	0.01651225	16512.25	1/32	838.29	15673.96	1.567×10^4
0.1443	0.02082249	20822.49	1/32	838.29	19984.20	1.998×10^4
0.1620	0.02624400	26244.00	1/32	838.29	25405.71	2.541×10^4
0.1819	0.03308761	33087.61	3/64	1886.15	31201.46	3.120×10^4
0.2043	0.04173849	41738.49	3/64	1886.15	39852.34	3.985×10^4
0.2294	0.05262436	52624.36	1/16	3353.15	49271.21	4.927×10^4
0.2576	0.06635776	66357.76	1/16	3353.15	63004.61	6.300×10^4
0.2893	0.08369449	83694.49	1/16	3353.15	80341.34	8.034×10^4
0.3249	0.10556001	105560.01	1/16	3353.15	102206.86	10.22×10^4
0.3648	0.13307904	133079.04	1/16	3353.15	129725.89	12.97×10^4
0.4096	0.16777216	167772.16	1/16	3353.15	164419.01	16.44×10^4
0.4600	0.21160000	211600.00	3/32	7544.60	204055.40	20.41×10^4

[a] The reduction in area due to the rounding of the corners.

TABLE II.—CALCULATED REDUCTION IN AREA IN SQUARE MILS FROM CIRCUMSCRIBING RECTANGLES DUE TO ROUNDING OF CORNERS OF RECTANGULAR WIRE.

Specified Thickness, in.	Specified Width, in.		
	0.751 and Over	0.189 to 0.750, incl	0.188 and Under
	Calculated reduction, sq mils		
0.689 and over	30178.38	30178.38	...
Under 0.689 to 0.439, incl.	13412.61	7544.60	...
Under 0.439 to 0.226, incl.	7544.60	3353.15	...
Under 0.226 to 0.166, incl.	3353.15	1886.15	1886.15
Under 0.166 to 0.126, incl.	3353.15	838.29	838.29
Under 0.126 to 0.073, incl.	[a]	838.29	209.57
Under 0.073 to 0.051, incl.	[a]	[a]	209.57
Under 0.051	[a]	[a]	[a]

[a] For wire with rounded edges, the calculated reduction in area in square mils is equivalent to 214600 T^2, where T is the thickness of the wire in inches. For square wire, see Table II (accompanying Table I).

equations should, for practical purposes, be rounded off to the number of significant figures shown in column 1. Attention is also called to the fact that the values obtained by these equations are for wire of nominal dimensions and do not take into account probable increase or decrease of the values due to the variations of the dimensions of an actual wire within the limits of the specified tolerances.

Table III.—Revise as shown in the accompanying Table II.

Note 8.—Revise to read as follows:

NOTE 8.—Table III (accompanying Table II) gives the calculated area in square mils to be deducted, because of the rounding of the four corners of the rectangular wire from the area of a circumscribing rectangle having the same thickness and width, in order to obtain the working net area of the wire. The areas to be deducted are based on the radii specified in Table I of these specifications, and do not take into account probable increase or decrease of the area of an actual wire due to the variation in its dimensions within the limits of the tolerances given in these specifications—as in the case of square wire, it has been demonstrated that there is no practical need for expressing working net areas of rectangular wire to a number of significant figures greater than that employed in specifying its thickness and width. This is also true of any other derived values such as circular mil area, weight or electrical resistance.

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[6]

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[6] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Editorial and Records (A. A. Jones, chairman) continued its editorial review of specifications and now is considering recommendations from the other subcommittees regarding inclusion of a "Basis of Purchase" section in the various Committee B-1 specifications.

Subcommittee II on Methods of Test and Sampling Procedure (J. B. Dixon, chairman) has completed its cooperative undertaking with Subcommittee IV on the revision of Specifications B 1 to include sampling procedures based on statistical methods, and now is engaged in developing similar revisions for Specifications B 3. Also, it is reviewing the adequacy of methods of determination of weights, areas and resistivities in all Committee B-1 specifications.

Subcommittee IV on Conductors of Copper and Copper Alloys (B. J. Sirois, chairman) completed the drafting of the proposed specifications for HD and MHD tinned copper wire and cooperated with Subcommittee II in the revision of Specifications B 1.

A task group has nearly completed its work on the development of a "Go-Not Go" gage for inspection testing of grooved trolley wire.

The task group on revision of significant figures in all tables of wire sizes has been collaborating with the National Bureau of Standards to establish bases of calculation to be used in the forthcoming revision of *Circular No. 31.* Agreement has been reached on basic wire sizes, and methods of computation of certain derived data.

Another task group is working on the preparation of proposed specifications covering the American Wire Gage. This work is well advanced, and it is hoped the proposal can be referred to the Society in the near future.

Other task groups are considering stiffness test requirements for magnet wire, the optimum percentage of tin in lead-alloy coatings, and possibly reduced tensile strength requirments for wire in coils of smaller than normal diameter.

Subcommittee V on Conductors of Ferrous Metals (L. H. Winkler, chairman) has a task group engaged in revising Specifications B 245 for steel core wire (for ACSR) having standard zinc coating, and another task group preparing proposed specifications for steel core wire having heavier than standard zinc coatings.

Subcommittee VI on Composite Conductors of Copper and Steel (C. E. Ambelang, chairman) has surveyed the possible need for a specification covering tinned copper-covered-steel wire. This survey indicated no need for such a specification at present, since only about one per cent of the copper-covered-steel wire produced is tinned, and this material usually will meet the same requirements as for uncoated wire.

Subcommittee VII on Conductors of Light Metals (P. V. Faragher, chairman) has a task group engaged in revising Specification B 233 for aluminum redraw rod, and another task group considering possible revisions in Specifications B 230 for aluminum wire.

This report has been submitted to letter ballot of the committee, which consists of 75 members; 61 members returned their ballots, all of whom have voted affirmatively.

Respectively submitted on behalf of the committee,

D. HALLORAN,
Chairman.

A. A. JONES,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee B-1 presented to the Society through the Administrative Committee on Standards the recommendation that Specifications for Standard Nominal Diameters and Cross-Sectional Areas of AWG Sizes of Solid Round Wires Used as Electrical Conductors be published as tentative. This recommendation was accepted by the Standards Committee on September 12, 1951, and the specifications appear in the 1951 Supplement to Book of ASTM Standards, Part 2, bearing the designation B 258 – 51 T.

REPORT OF TASK GROUP ON SEGMENTAL CONDUCTORS OF SUBCOMMITTEE IV

This Task Group has made several attempts to write a satisfactory and all-inclusive specification for segmental conductors, and has distributed these for comment. On January 5, 1951, the Task Group met and reviewed the problem in view of the comments and data available at this time. The situation may be summarized as follows:

1. *Design.*—The design of segmental conductors is still in the experimental stage and the design is influenced both by the end use of the cable and the manufacturing equipment available.

2. *Number of Segments.*—Some manufacturers and users favor three segments, some four, and hollow cables of both four and six segments have been made.

3. *Construction of Segments.*—The individual segments may be formed by using strands of different diameter or by crushing or compacting by means of rolls, and the stranding may be unilay or reverse lay. The number of strands used is influenced by the method of forming.

4. *Coating of Wires.*—On constructions used with rubber or rubber-like insulations, some manufacturers use tinned or alloy-coated conductors; others use coated copper binder tapes over the assembly.

5. *Treatment Prior to Insulating.*—For use with certain types of insulation some manufacturers treat the strands to produce an oxide coating. This practice is not universal.

6. *Insulation of Individual Segments.*—There is a wide difference of opinion in regard to taping of segments. Usually two segments are taped, sometimes three. One or two tapes may be used. To insure that there is no metal-to-metal contact between segments, an electrical test is frequently required. The number of tapes and whether they are dry or impregnated affects the test values.

7. *Binder Tape.*—Binder tape practice varies with the type of insulation and the manufacturer. For example, conductors for use with rubber insulation may have a semi-conducting fabric tape binder or a metal tape. On other applications a lapped tape or an intercalated tape may be used.

8. *Conductor Diameters.*—The Task Group has been able to establish a tabulation of conductor diameters of several typical constructions which are considered sufficiently accurate for estimating overall cable diameters and sheath thicknesses. At the present time, however there is little prospect of being able to establish diameters under binder tapes with sufficient accuracy for use in design or standardization of connectors. Appendix I attached gives approximate diameters for several constructions which may be of value for estimating purposes. They should be used with caution for the reasons indicated above.

9. *AC/DC Ratios.*—It does not seem practical at this time to set up AC/DC ratios for general design purposes as there is not sufficient test data available to enable one to distinguish with certainty between acceptable designs and those that are undesirable.

SUMMARY

In view of the above situation it is evident that the design of segmental conductors is still in the experimental stage, with varied ideas on construction and insufficient experimental data or field experience to establish the general acceptance of any design.

Inasmuch as segmental conductors are rarely, if ever, used other than as a component of an insulated cable design, and inasmuch as the conductor itself, having some segments insulated, may be considered as a type of insulated conductor assembly, it would seem that, for the present at least, the design details of the conductor be covered by the insulated cable specification of which they are a part.

If in the future a certain specific design appears generally acceptable, as in the somewhat parallel case of annular conductors, a specification can be written for this construction.

Submitted by Task Group on Segmental Conductors, Subcommittee IV, ASTM Committee B-1:

G. W. Zink, chairman,
C. A. Finkel,
D. Halloran,
A. H. Kidder,
M. H. McGrath, and
K. D. Roberts.

APPENDIX I

The following table includes typical segmental conductors in common use. The table is not all-inclusive, but does illustrate certain constructions.

Size, cir mils	Number of Strands per Segment	Minimum Total Number of Wires	Nominal Diameter over Binder, in.	D-C Resistance, ohms per 1000 ft	
				25 C	65 C
SEGMENTAL CONDUCTORS COMPOSED OF FOUR SEGMENTS COMPACT TYPE					
1 000 000	37	148	1.15	0.0108	0.0125
1 250 000	37	148	1.29	0.00867	0.0100
1 500 000	37	148	1.41	0.00723	0.00834
1 750 000	37	148	1.53	0.00619	0.00714
2 000 000	61	244	1.63	0.00542	0.00626
2 500 000	61	244	1.83	0.00434	0.00501
3 000 000	91	364	2.00	0.00361	0.00417
3 500 000	91	364	2.16	0.00310	0.00358
4 000 000	91	364	2.31	0.00271	0.00313
SEGMENTAL CONDUCTORS COMPOSED OF THREE SEGMENTS COMPACT TYPE					
1 000 000	37	111	1.25	0.0108	0.0125
1 250 000	37	111	1.33	0.00867	0.0100
1 500 000	61	183	1.45	0.00723	0.00834
1 750 000	61	183	1.58	0.00619	0.00714
NON-COMPACT TYPE					
1 000 000	51	153	1.30	...	...
1 250 000	51	153	1.41	...	...
1 500 000	51	153	1.56	...	...
1 750 000	51	153	1.69	...	...

REPORT OF TASK GROUP ON TROLLEY WIRE OF SUBCOMMITTEE IV

Partly in view of long-standing controversy and particularly in view of certain minor dimensional changes made in certain sizes of trolley wire the preceding year, this Task Group was instructed in the Spring of 1949 to recalculate at least a representative number of the trolley wire areas to determine whether or not the areas as published were correct. Recomputation of 5 sizes of grooved wire indicated a maximum error of 0.2 per cent, which is considered satisfactory accuracy. Therefore, no additional computations, and no changes in the published data will be made. For the record, the results are included below:

AREAS AND WEIGHTS OF GROOVED TROLLEY WIRE.

Nominal Size, cir. mils	Area, cir mils	Area, sq in.	Weight, lb per mile
FROM SPECIFICATIONS B 9 AND B 47			
350 000	351 200	0.2758	5 612
300 000	299 800	0.2355	4 792
211 600	212 000	0.1665	3 389
168 100	167 300	0.1314	2 674
133 200	137 900	0.1083	2 205
FROM CALCULATIONS			
350 000	350 649	0.2754	5 604
300 000	300 229	0.2358	4 798
211 600	211 993	0.1665	3 388
168 100	167 303	0.1314	2 674
133 200	137 636	0.1081	2 199

Submitted by Task Group on Trolley Wire, Subcommittee IV, ASTM Committee B-1:

L. B. Curtis, chairman.
W. T. Myers,
R. S. Pratt,
B. J. Sirois,
L. F. Brahmer,
L. W. Birch, and
A. A. Jones.

REPORT OF COMMITTEE B-3

ON

CORROSION OF NON-FERROUS METALS AND ALLOYS*

Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys has held no meeting during the past year.

The committee consists of 106 members of whom 85 are voting members; 28 are classified as consumers, 37 as producers, and 20 as general interest members.

At a meeting of Committee B-3 on June 21, 1951 during the Annual Meeting of the Society, Dr. Henry S. Rawdon was elected Honorary Chairman of the committee. Dr. Rawdon contributed many years service to committee activities. He served two terms as chairman and took active part in many subcommittee programs.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends that the three test methods under its jurisdiction be continued as tentative because of possible revisions from work planned:

Tentative Methods of Tests for:

Total Immersion Corrosion Testing of Non-Ferrous Metals (B 185 - 43 T),
Salt Spray Fog Testing (B 117 - 49 T), and
Alternate Immersion Corrosion Test of Non-Ferrous Metals (B 192 - 44 T).

ACTIVITIES OF SUBCOMMITTEES

Subcommittee III on Spray Testing (N. L. Foltz, chairman).—A work group of this subcommittee is studying an acetic acid - salt spray test for evaluation of plated coatings on zinc-base die castings.

Subcommittee VI on Atmospheric Corrosion (W. H. Finkeldey, chairman).—All the remaining specimens on exposure at Phoenix, Ariz., and Key West, Fla., on test since 1931, have been removed from the test racks. The site at Phoenix on the property of the Central Arizona Light and Power Co. was needed for plant expansion and the test station has been abandoned. The specimens at Key West were removed after inspection by the chairman of Committee B-3.

This report has been submitted to letter ballot of the committee, which consists of 85 voting members; 50 returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

K. G. COMPTON,
Chairman.

A. W. TRACY,
Secretary.

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* Presented at the Fifty-fourth Annual Meeting of the Society, June 18-22, 1951.

REPORT OF COMMITTEE B-4

ON

ELECTRICAL HEATING, RESISTANCE, AND RELATED ALLOYS*

Committee B-4 on Electrical Heating, Resistance, and Related Alloys has held three meetings during the year: in Philadelphia, Pa., on June 6 and 7, 1950; in Philadelphia, on October 26 and 27, 1950; in New York, N. Y., on March 1 and 2, 1951.

In the death of F. E. Bash, Committee B-4 lost one of its very active members. Mr. Bash was secretary of Committee B-4 for over 25 years. The committee wishes to record its appreciation of his many contributions to its work.

The present membership of this committee totals 72 members, of whom 30 are classified as producers, 33 as consumers, and 9 as general interest members.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Electrical Heating Materials (C. L. Raynor, chairman) has concentrated on reconsideration of existing specifications. The Standard Method of Accelerated Life Test for Metallic Materials for Electrical Heating (B 76 – 39), has been studied and revisions in the temperature sections are currently out to letter ballot of B-4. This subcommittee has been concerned with the new design of test chambers for accelerated life tests. In addition, improvements in the terminology used in this specification are being considered. Specifications B 82 – 46 and B 83 – 46, concerning alloys drawn or rolled for electrical heating elements, also are under re-examination, particularly on the basis of the controls of wire sizes and resistance.

Subcommittee V on Wrought and Cast Alloys for High Temperatures Use (C. L. Raynor, chairman).—C. L. Raynor replaced R. Van Nordstrand as chairman of this subcommittee upon Mr. Van Nordstrand's retirement.

Subcommittee VII on Thermostat Metals (P. H. Brace, chairman).—Modifications of thickness-width ratios, as specified in the Standard Method of Testing Thermostat Metals (B 106 – 40), to coincide more exactly with standard test samples commonly used in the industry are under consideration. Subcommittee VII is also concerning itself with test methods and specifications for invar and related low expansivity alloys.

Subcommittee X on Contact Materials (F. E. Carter, chairman) has five active sections dealing with the problems of metallic contacts. The current work load of this subcommittee includes life tests, improved methods of determining surety of making contact, and the effect of oxidation and sulfidation treatment on contacts. The section on Physical Properties has completed its method of determining hardness of contacts and a proposed specification has been referred to Committee E-1 for comment. In addition to the above projects the section on Contact Forms has tentatively tabulated the forms most commonly used, keeping in mind that the aim is to settle on a minimum number of dimensions to cover as broad a field as possible.

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* Presented at the Fifty-fourth Annual Meeting of the Society, June 18–22, 1951.

The section on Bibliography on Contacts has decided to revise the "Bibliography on Electrical Contacts" to include all references up to and including 1950. A new section on Micro-current Contacts has been formed.

Subcommittee VIII on Metallic Materials for Radio Tubes and Incandescent Lamps (S. A. Standing, chairman).—Section A, concerned with specifications on nickel used for oxide-coated cathodes, has summarized and circulated the report on five years' data of commercial "normal" grade cathode melt tests by the industry. During the past year the ground work for improving present diode test tubes has received considerable attention pointing to revision and possible adoption of a triode structure. These activities are augmented by gas analysis work and broader chemical and spectrochemical analysis approaches. Several methods for small sample chemical analysis of cathode "impurities" have been approved in cooperation with Committee E-3 and, when others are completed, a broad specification will be set forth. This section has also cooperated with the Joint Electron Tube Engineering Council (JETEC) in preparing a summary of cathode materials and sizes in connection with programs concerning strategic materials.

Section B was set up to cover the testing of fine wires smaller than 0.005 in. in diameter. For the most part the work of this group is to develop methods for determining tungsten fine wire diameters by weighing and to seek means of improving the Standard Method of Test for the Density of Fine Wire and Ribbon for Electronic Devices (B 180 – 50).

Section E has developed specifications for a suitable metal for metal-to-glass seals on television picture tubes. These specifications, one for a 17 per cent chromium-iron alloy and another for a 28 per cent chromium-iron alloy, will be submitted to the Administrative Committee on Standards.

Section F on Measurement of Mica Stampings revised the Standard Method of Test for Measurement of Mica Stampings Used in Electronic Devices and Incandescent Lamps (D 652 – 43) in the light of more current industry needs. This group completed its assignment to the end of sending the revision to Committee D-9 on Electrical Insulating Materials under whose jurisdiction Methods D 652 fall.

Respectfully submitted on behalf of the committee,

S. A. STANDING,
Chairman.

STANTON UMBREIT,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee B-4 presented to the Society through the Administrative Committee on Standards the following recommendations:

Tentative Specifications for:

17 Per Cent Chromium Iron Alloy for Sealing to Glass (B 256 – 51 T), and
28 Per Cent Chromium Iron Alloy for Sealing to Glass (B 257 – 51 T).

These recommendations were accepted by the Standards Committee on July 10, 1951, and the specifications appear in the 1951 Supplement to Book of ASTM Standards, Part 2.

On August 21, 1951, the Administrative Committee on Standards accepted the following recommendations submitted by Committee B-4:

Revision and Reversion to Tentative of:

Standard Methods of Testing Thermostat Metals (B 106 – 40), and
Standard Method of Test for Modulus of Elasticity of Thermostat Metals (Cantilever Beam Method) (B 223 – 50).

The revised methods appear in the Supplement to 1951 Book of ASTM Standards, Part 2.

REPORT OF COMMITTEE B-5

ON

COPPER AND COPPER ALLOYS, CAST AND WROUGHT*

Committee B-5 on Copper and Copper Alloys, Cast and Wrought, held two meetings during the year: at New York, N. Y., on September 28, 1950, and at Philadelphia, Pa., on January 31, 1951. At these sessions the Advisory Committee and Subcommittees W-1, W-2, W-3, W-4, F-1, G-1 and G-3 also met.

During the year 17 members were added to the committee and there were 15 removals. At the present time the committee consists of 136 members, of whom 115 are voting members; 52 are classified as producers, 44 as consumers, and 19 as general interest members.

RECOMMENDATIONS AFFECTING STANDARDS

The committee is submitting 4 new tentative specifications, revisions in 20 tentative specifications, revisions in 20 standards for immediate adoption, the adoption of 3 tentatives as standard, the adoption of 5 tentative revisions as standard, and the withdrawal of 1 tentative. The standards and tentatives affected, together with the revisions recommended, are covered in detail in the Appendix.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[1]

ACTIVITIES OF THE ADVISORY COMMITTEE

It was recommended that the Tentative Classification of Coppers, B 224, be adopted as standard.

ACTIVITIES OF SUBCOMMITTEES

The following is the report of the several subcommittees during the year. The actions on standards are given in detail in the Appendix.

Subcommittee W-1 on Plate, Sheet, and Strip (S. A. Rosecrans, chairman, W. D. France, secretary) recommended that revisions be incorporated in the following eight tentative specifications:

ASTM Designation	Material
B 19.......	Cartridge Brass
B 36.......	Brass
B 121......	Leaded Brass
B 122......	Copper-Nickel-Zinc and Copper-Nickel Alloy
B 130......	Gilding Metal
B 152......	Copper
B 169......	Aluminum Bronze
B 194......	Beryllium-Copper Alloy

Revisions for immediate adoption were recommended in four standard specifications:

ASTM Designation	Material
B 96.......	Copper-Silicon Alloy for Pressure Vessels
B 97.......	Copper-Silicon Alloy for General Purposes
B 103......	Phosphor Bronze
B 171......	Copper-Alloy Condenser Tube Plates

Specifications B 129 and B 131 were recommended for adoption as standard. It was recommended that Specifications

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* Presented at the Fifty-fourth Annual Meeting of the Society, June 18-22, 1951.

[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

B 195 on Beryllium-Copper Alloy Strip, Special Grade, be withdrawn since the material would be covered in the proposed revisions for Specifications B 194. The subcommittee prepared a proposed new Tentative Specification for General Requirements for Wrought Copper and Copper-Alloy Plate, Sheet, Strip and Rolled Bar.

A task group (H. C. Ashley, chairman) prepared requirements for aluminum bronze, alloy D, to be incorporated in Specifications B 171. A task group (W. F. Roeser, chairman) prepared revisions of Rockwell hardness information in eight plate, sheet, and strip specifications. A task group (H. S. Freynik, chairman) prepared revisions in chemical and mechanical test requirements in Specifications B 194. A task group (V. P. Weaver, chairman) prepared revisions in chemical requirements for alloy A and alloy A-7 in Specifications B 96 and B 97. A task group (R. S. Pratt, chairman) prepared the new Tentative Specification for General Requirements for Wrought Copper and Copper-Alloy Plate, Sheet, Strip and Rolled Bar, and the accompanying revisions of Specifications B 36, B 97, B 103, B 121, B 122, B 152, B 169, and B 194.

Among the subjects being studied by the subcommittee are the addition of sawed bar to plate specifications, the addition of alloy No. B2 of Specifications B 139 for Phosphor Bronze Rods, Bars, and Shapes to Specifications B 103, limitations of thickness for Rockwell hardness, the preparation of requirements for thicker plates in Specifications B 171 and the preparation of yield strength data for material covered by Specifications B 11, used by ASME.

Subcommittee W-2, Rods, Bars, and Shapes (J. D. MacQueen, chairman) recommended that revisions be incorporated in the following six tentative specifications:

ASTM Designation	Material
B 21	Naval Brass
B 133	Copper
B 139	Phosphor Bronze
B 150	Aluminum Bronze
B 151	Copper-Nickel-Zinc Alloy
B 196	Beryllium-Copper Alloy

Revisions for immediate adoption were recommended in five standard specifications:

ASTM Designation	Material
B 16	Free-Cutting Brass
B 98	Copper-Silicon Alloy
B 124	Copper and Copper-Base Alloy Forging Rods
B 138	Manganese Bronze
B 140	Leaded Red Brass (Hardware Bronze)

Specifications B 187 were recommended for adoption as standard. The subcommittee prepared a proposed new Tentative Specification for General Requirements for Wrought Copper and Copper-Alloy Rod, Bar, and Shapes. Adoption as standard was recommended for tentative revisions of five standard specifications: B 12, B 16, B 98, B 138, and B 140.

A task group (J. E. McGraw, chairman) prepared the revisions of tensile properties for hard temper rods and bars of alloy A in Specifications B 138. A task group (V. P. Weaver, chairman) prepared the revisions of chemical requirements for alloys A and A-7 in Specifications B 98. A task group (H. S. Freynik, chairman) prepared the revisions of chemical composition and mechanical test requirements in Specifications B 196. A task group (H. C. Ashley, chairman) prepared the new Tentative Specification for General Requirements for Wrought Copper and Copper-Alloy Rod, Bar, and Shapes, and the accompanying revisions of Specifications B 16, B 21, B 98, B 124, B 133, B 138, B 139, B 140, B 150, B 151 and B 196.

Among the subjects being considered in the subcommittee are the preparation of specifications for forgings, review of tensile requirements in Specifications B 133, the hardness testing of rods and bars in Specifications B 16, B 133, B 187, and B 196, and the preparation of yield strength data for the material covered by Specifications B 12 and B 98, used by ASME.

Subcommittee W-3 on Wire and Wire Rod (E. W. Lovering, chairman) recommended that revisions be incorporated in three tentative specifications: B 159 for Phosphor Bronze, B 197 for Beryllium-Copper Alloy, and B 206 for Copper-Nickel-Zinc Alloy. Revisions for immediate adoption were recommended in Standard Specifications B 99 for Copper-Silicon Alloy and B 134 for Brass. The subcommittee prepared a proposed new Tentative Specification for General Requirements for Wrought Copper-Alloy Wire.

A task group (G. R. Gohn, chairman) prepared the new Tentative Specification for General Requirements for Wrought Copper Alloy Wire and accompanying revisions of Specifications B 99, B 134, B 159, B 197 and B 206.

Among the subjects being studied by the subcommittee are the preparation of new specifications for rectangular copper wire and the development of requirements for flat wire in Specifications B 159, B 197, and B 206.

Subcommittee W-4 on Pipe and Tube (R. S. Pratt, chairman) recommended that revisions be incorporated in three tentative specifications: B 75 for Copper, B 135 for Brass, and B 188 for Copper Bus Pipes and Tubes. Revisions for immediate adoption were recommended in the following five standard specifications:

ASTM Designation	Material
B 42	Copper Pipe
B 43	Red Brass Pipe
B 68	Bright Annealed Copper
B 88	Copper Water Tube
B 111	Condenser Tubes

The subcommittee prepared a proposed new Tentative Specification for General Requirements for Wrought Seamless Copper and Copper-Alloy Pipe and Tube.

A task group (V. P. Weaver, chairman) prepared the revisions of Specifications B 111 to provide for light-drawn temper for 90–10 copper-nickel alloy. A task group (J. E. McGraw, chairman) prepared the proposed new Tentative Specification for General Requirements for Wrought Seamless Copper and Copper-Alloy Pipe and Tube, and the accompanying revisions in Specifications B 42, B 43, B 68, B 75, B 88, B 135 and B 188.

Among the subjects under consideration in the subcommittee are the development of requirements for refrigeration service tube, review of temper requirements in Specifications B 75 for Seamless Copper Tubes, and review of Rockwell hardness requirements in tube specifications. For Specifications B 111, the subcommittee is studying the revision of dimensional tolerances and requirements for iron content in 70–30 copper-nickel alloy. Yield strength data are being prepared for the following specifications used by ASME: B 13, B 42, B 43, B 75 and B 111.

Subcommittee F-1 on Castings, and Ingots for Remelting (G. H. Clamer, chairman, M. L. Steinbuch, secretary) recommended revisions for immediate adoption in three standard specifications: B 22, B 61, and B 62.

A task group (J. W. Bolton, chairman) prepared the complete revisions of Specifications B 61 and B 62 to cover component castings of valves, flanges, and fittings.

Among the subjects under consideration in the subcommittee are the preparation of a document similar to British Standard 1367: 1947, "Code of Procedure in Inspection of Copper-Base Alloy

Sand Castings," the preparation of specifications for a group of copper-nickel-tin-zinc alloys, and preparation of specifications for centrifugal castings. The subcommittee is also reviewing the Tentative Recommended Practice for Tension Test Specimens for Copper-Base Alloys for Sand Castings, B 208, as to whether it should be recommended for adoption as standard.

Subcommittee G-1 on Methods of Test (G. R. Gohn, chairman, J. P. Guerard, secretary) recommended revisions for immediate adoption in the Standard Method of Mercurous Nitrate Test for Copper and Copper Alloys (B 154 – 50) as prepared by a task group (N. H. Murdza, chairman).

The subcommittee has completed its studies on the effect of speed of testing on tensile properties and found that, within the ability of commercially available testing machines to accurately record load and strain, there is no significant effect due to speed of test on tensile strength, yield strength or per cent elongation of copper and copper alloys. A paper covering the results of these studies is being prepared and will be presented before the Society at a later session.

Since the Tentative Methods of Tension Testing of Copper and Copper-Alloy Rods, Bars, and Shapes (B 220 – 48 T) is being combined with the Tentative Methods of Tension Testing of Metallic Materials (E 8 – 51T),[2] the subcommittee is considering the desirability of discontinuing the former method of test.

The subcommittee is engaged in the preparation of a method of test covering the measurement of dimensions, such as thickness, width, diameter and length. Also, a task group (G. H. Harnden, chairman) is reviewing the previous studies on the determination of grain size, and plans to issue a report in the near future; several new grain size standards will be recommended.

Subcommittee G-2 on Tolerances (J. E. McGraw, chairman) in cooperation with Subcommittee W-1, is preparing dimensional tolerances for thicker plates in Specifications B 171.

For all wrought product specifications prepared by Committee B-5, the subcommittee is reviewing dimensional tolerances as to permitted deviation.

Subcommittee G-3 on Editorial and Publications (W. F. Roeser, chairman) recommended revision of the section on Workmanship and Finish in wrought product specifications.

The subcommittee is continuing the study of nomenclature and classification of wrought alloys under the jurisdiction of the committee and is also preparing a more detailed index of the contents of the volume of ASTM Standards on Copper and Copper Alloys.

This report has been submitted to letter ballot of the committee, which consists of 115 voting members; 93 members returned their ballots, of whom 90 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

G. H. HARNDEN,
Chairman.

V. P. WEAVER,
Secretary.

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[2] 1951 Supplement to Book of ASTM Standards, Part 2.

APPENDIX

RECOMMENDATIONS AFFECTING STANDARDS FOR COPPER AND COPPER ALLOYS, CAST AND WROUGHT

In this Appendix are given the recommendations affecting certain standards covering copper and copper alloys which are referred to earlier in this report. The standards appear in their present form in the 1949 Book of ASTM Standards, Part 2, or in the 1950 Supplement to the Book of ASTM Standards, Part 2.

In addition to the revisions outlined on the following pages, all of the specifications shall have their titles changed from theplural. "Specifications" to the singular "Specification." Also the "Basis of Purchase" sections will be editorially revised to bring the references into line with the new coverage by the applicable General Requirement Specification explained under the section on "New Tentatives."

It is expected that other editorial changes may be found necessary in the product specifications in order to co-ordinate them with the several General Requirement Specifications.

NEW TENTATIVES

The committee recommends the following four new specifications for publication as tentative:[1]

Tentative Specifications for:[1]

General Requirements for Wrought Copper and Copper-Alloy Plate, Sheet, Strip, and Rolled Bar;

General Requirements for Wrought Copper and Copper-Alloy Rod, Bar, and Shape;

General Requirements for Wrought Copper-Alloy Wire; and

General Requirements for Wrought Seamless Copper and Copper-Alloy Pipe and Tube.

These specifications consist of requirements common to the field of copper and copper-alloy products covered, which requirements are recommended to be deleted from the individual commodity specifications in the following sections of this Appendix. It is recommended that these General Requirement Specifications be accepted for publication as tentative.

REVISIONS OF TENTATIVE SPECIFICATIONS

The committee recommends that the following 20 tentative specifications be revised as indicated below and continued as tentative.

Tentative Specifications for Cartridge Brass Sheet, Strip, Plate, Bar and Disks (B 19 – 50 T):

Table I.—Revise title to read: "Tensile Strength Requirements and Approximate Rockwell Hardness Values for Rolled

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[1] The new tentatives were accepted by the Society and appear in the 1951 Supplement to Book of ASTM Standards, Part 2.

Tempers." Revise the third column heading to read "Approximate Rockwell Hardness[a]," retaining the present footnote *a*.

Section 9.—Delete Paragraph (*b*).

Section 10.—Revise to read: "Since Rockwell hardness tests offer a quick and convenient method of checking cartridge brass of any temper for general conformity to the requirements for tensile strength, the approximate Rockwell hardness values for each of the rolled tempers are given in Table I for general information and assistance in testing."

Table IV.—For Type IV, revise the present minimum grain size "0.055" to read "0.075."

Tentative Specifications for Naval Brass Rods, and Shapes (B 21 – 50 T):

New Section.—Add a new Section 2 to read as follows, renumbering subsequent sections accordingly:

> 2. *General Requirements.*—Material furnished under this specification shall conform to the applicable requirements of the current edition of the Tentative Specification for General Requirements for Wrought Copper and Copper Alloy Rod, Bar, and Shapes (ASTM Designation: B—).[2]

Sections 3, 5, 9, 10 and 11.—Delete and renumber subsequent sections accordingly.

Section 12.—Renumber as Section 8 and revise to read as follows:

> 8. *Dimensions and Tolerances* (*a*) The dimensions and tolerances for material covered by this specification shall be as prescribed in the Tentative Specification for General Requirements for Wrought Copper and Copper Alloy Rod, Bar, and Shapes (ASTM Designation: B—),[2] with particular reference to Sections 8 and 9 and the following tables of that specification.
>
> (*b*) *Diameter or Distance Between Parallel Surfaces:*
>
> (*1*) *Rod; Round, Hexagonal, Octagonal.*—See Table I.
>
> (*2*) *Piston Finish Rod.*—See Table III.
>
> (*3*) *Bar; Rectangular and Square.*—See Tables VIII and X.
>
> (*c*) *Shape.*—The dimensional tolerances for shape shall be as agreed upon by the manufacturer and the purchaser, and shall be specified in the order.
>
> (*d*) *Length of Rod, Bar, and Shapes.*—See Section 8(*c*), Tables XII and XIII.
>
> (*e*) *Straightness:*
>
> (*1*) *Rod, Bar.*—See Section 9(*a*) and Table XV.
>
> (*2*) *Shafting Rod.*—See Section 9(*b*) and Table XVI.

Section 13.—Delete and renumber subsequent sections accordingly.

Section 14.—Delete Paragraph (*c*).

Sections 15, 16, 17 and 18.—Delete.

Tentative Specifications for Brass Plate, Sheet, and Strip (B 36 – 50 T):

New Section.—Add a new Section 2 to read as follows, renumbering subsequent sections accordingly:

> 2. *General Requirements.*—Material furnished under this specification shall conform to the applicable requirements of the current edition of the Tentative Specification for General Requirements for Wrought Copper and Copper-Alloy Plate, Sheet, Strip and Rolled Bar (ASTM Designation: B—).[2]

Sections 3 and 5.—Delete and renumber subsequent sections accordingly.

Section 8.—Delete Paragraph (*b*).

Table I.—Revise title to read "Tensile Strength Requirements and Approximate Rockwell Hardness Values for Rolled Tempers." Revise the third column heading to read: "Approximate Rockwell Hardness[a]," retaining the present footnote *a*.

Section 9.—Delete and renumber subsequent sections accordingly.

Section 11.—Renumber as Section 9 and revise to read as follows:

> 9. *Rockwell Hardness.*—Since Rockwell hardness tests offer a quick and convenient method of checking brass of any temper for general

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[2] See p. 151, under "New Tentatives," for an explanation of these specifications.

conformity to the requirements for tensile strength or grain size, the approximate Rockwell hardness values for each temper are given in Table I or III for general information and assistance in testing.

Table III.—Revise the second column heading to read "Approximate Rockwell Hardness[a]," retaining the present footnote *a*.

Sections 12, 13, and 14.—Delete and renumber subsequent sections accordingly.

Section 15.—Renumber as Section 10 and revise to read as follows:

> 10. *Dimensions and Tolerances* (*a*) The dimensions and tolerances for material covered by this specification shall be as prescribed in the Tentative Specification for General Requirements for Wrought Copper and Copper-Alloy Plate, Sheet, Strip and Rolled Bar (ASTM Designation: B—),[2] with particular reference to Section 8 and the following tables of that specification.
>
> (*b*) *Thickness.*—See Table I.
>
> (*c*) *Width:*
>
> (*1*) *Slit-Edge and Rolled-Edge Strip.*—See Table IV.
>
> (*2*) *Square-Sheared-Edge Metal.*—See Table V.
>
> (*3*) *Sawed-Edge Metal.*—See Table VI.
>
> (*d*) *Length:*
>
> (*1*) *Specific and Stock Lengths.*—See Table VII.
>
> (*2*) *Schedule of Lengths* (*Specific and Stock*) *with Ends.*—See Table VIII.
>
> (*e*) *Straightness:*
>
> (*1*) *Slit-Edge Strip.*—See Table IX.
>
> (*2*) *Rolled-Edge Strip.*—See Table X.
>
> (*3*) *Square-Sheared-Edge Metal.*—See Table XI.
>
> (*4*) *Sawed-Edge Metal.*—See Table XII.

Sections 16, 17, 18, 19 and Appendix.—Delete.

Tentative Specifications for Seamless Copper Tubes (B 75 - 49 T):

Section 1.—Revise the second sentence to read "Four types may be specified as indicated below." Add a third sentence to read "Unless otherwise specified, any one of these types may be supplied."

New Section.—Add a new Section 2 to read as follows, renumbering subsequent sections accordingly.

> 2. *General Requirements.*—Material furnished under this specification shall conform to the applicable requirements of the current edition of the Tentative Specification for General Requirements for Wrought Seamless Copper and Copper-Alloy Pipe and Tube (ASTM Designation: B—).[2]

Section 2.—Revise Item (*1*) to read "Type of copper, if required (Sections 1, 4)."

Sections 3, 5, 13, 14, 15 and 16.—Delete and renumber subsequent sections accordingly.

Section 9.—Add a new paragraph (*a*) to read as follows, relettering subsequent paragraphs accordingly:

> (*a*) For the purpose of hydrostatic testing only, a number of tubes equivalent to 0.2 per cent of the shipment shall be selected at random for the following test.

Section 17.—Renumber as Section 12 and revise to read as follows:

> 12. *Dimension and Tolerances* (*a*) The dimension and tolerances for material covered by this specification shall be as prescribed in the Tentative Specification for General Requirements for Wrought Seamless Copper and Copper-Alloy Pipe and Tube (ASTM Designation: B—),[2] with particular reference to Section 9 and the following tables of that specification.
>
> (*b*) *Wall Thickness.*—See Section 9(*d*), Table VI.
>
> (*c*) *Diameter.*—See Section 9(*e*), Table VII.
>
> (*d*) *Length.*—See Section 9(*h*), Tables IX, X, and XI.
>
> (*e*) *Roundness.*—See Section 9(*f*).
>
> (*f*) *Squareness of Cut.*—See Section 9(*i*).
>
> (*g*) *Straightness.*—See Section 9(*j*).

Sections 18, 19, 20 and Appendix.—Delete.

Tentative Specifications for Leaded Brass Plate, Sheet, and Strip (B 121 - 50 T):

New Section.—Add a new Section 2 to read as indicated above for new

Section 2 in Specifications B 36, renumbering subsequent sections accordingly.

Sections 3 and 5.—Delete and renumber subsequent sections accordingly.

Section 8.—Delete Paragraph (*b*).

Table I.—Revise title to read: "Tensile Strength Requirements and Approximate Rockwell Hardness Values for Rolled Tempers." Revise the third column heading to read "Approximate Rockwell Hardness[a]," and revise footnote *a* to read "Rockwell hardness values apply as follows:"

Section 9.—Delete and renumber subsequent sections accordingly.

Section 11.—Renumber as Section 9 and revise to read as follows:

> 9. *Rockwell Hardness.*—Rockwell hardness tests offer a quick and convenient method of checking leaded brass of any temper for general conformity to the requirements for tensile strength or grain size. The approximate Rockwell hardness values for the rolled tempers are given in Table I, and those for the annealed tempers of material 0.015 in. and over in thickness are given in Table III for general information and assistance in testing.

Table III.—At the beginning of the title insert the word "Approximate," and revise the second column heading to read "Approximate Rockwell Hardness[a]." Revise footnote *a* to read "Rockwell hardness values apply as follows:".

Sections 12, 13, and 14.—Delete and renumber subsequent sections accordingly.

Section 15.—Revise to read as indicated above for Section 15 in Specifications B 36.

Sections 16, 17, 18, 19 and Appendix.—Delete.

Tentative Specifications for Copper-Nickel-Zinc and Copper-Nickel Alloy Plate, Sheet, and Strip (B 122 – 50 T):

New Section.—Add a new Section 2 to read as indicated above for new Section 2 in Specifications B 36, renumbering subsequent sections accordingly.

Sections 3 and 5.—Delete and renumber subsequent sections accordingly.

Section 8.—Delete Paragraph (*b*).

Table II.—Revise as indicated above for Table I in Specifications B 121.

Section 9.—Delete and renumber subsequent sections accordingly.

Section 11.—Renumber as Section 9 and revise to read as follows:

> 9. *Rockwell Hardness.*—Rockwell hardness tests offer a quick and convenient method of checking copper-nickel-zinc and copper-nickel alloys of any temper for general conformity to the requirements for tensile strength or grain size. The approximate Rockwell hardness values for the rolled tempers are given in Table II and those for the annealed tempers of material 0.015 in. and over in thickness are given in Table III for general information and assistance in testing.

Table III.—Revise the second column heading to read "Approximate Rockwell Hardness[a]," and revise footnote *a* to read "Rockwell hardness values apply as follows:".

Sections 12, 13, and 14.—Delete and renumber subsequent sections accordingly.

Section 15.—Revise to read as indicated above for Section 15 in Specifications B 36 except that "Table I" should read "Table II."

Sections 16, 17, 18, 19 and Appendix.—Delete.

Tentative Specifications for Gilding Metal Strip (B 130 – 50 T):

Section 9.—Delete Paragraph (*b*).

Table I.—Revise title to read "Tensile Strength Requirements and Approximate Rockwell Hardness Values for Cold-Rolled Gilding Metal Strip." Revise the third column heading to read "Approximate Rockwell Hardness[a]," retaining the present footnote *a*.

Section 10.—Revise to read "Since Rockwell hardness tests offer a quick and convenient method of checking gilding metal of any temper for general conformity to the requirements for tensile strength, the approximate Rockwell hardness values for each of the cold-rolled tempers are given in Table I for general information and assistance in testing."

Tentative Specifications for Copper Rods, Bars, and Shapes (B 133 - 49 T):

New Section.—Add a new Section 2 to read as indicated above for new Section 2 in Specifications B 21, renumbering subsequent sections accordingly.

New Section.—Add a new Section 5 to follow the present Section 4 on "Material" to read as follows, renumbering subsequent sections accordingly:

5. *Requirements for Shapes.*—Shapes shall conform to all requirements of this specification except temper (Sections 6 and 7) and dimensional tolerances (Section 13) for which special agreement shall be made between the manufacturer and the purchaser.

Sections 5, 6, 14, 15 and 16.—Delete and renumber subsequent sections accordingly.

Section 18.—Renumber as Section 13 and revise to read as follows:

15. *Dimensions and Tolerances* (*a*) The dimensions and tolerances for material covered by this specification shall be as prescribed in the Tentative Specification for General Requirements for Wrought Copper and Copper Alloy Rod, Bar, and Shapes (ASTM Designation: B—),[2] with particular reference to Sections 8 and 9 and the following tables of that specficaition.

(*b*) *Diameter or Distance Between Parallel Surfaces:*

(*1*) *Rod; Round, Hexagonal, Octagonal.*—See Table I.

(*2*) *Bar; Rectangular and Square.*—See Tables VII and X.

(*c*) *Shapes.*—The dimensional tolerances for shapes shall be as agreed upon by the manufacturer and the purchaser and shall be specified in the order.

(*d*) *Rolls.*—Unless otherwise specified, soft temper bar shall be furnished in rolls. The minimum roll weight shall be 150 lb. The inside diameter of the roll shall be not less than 18 in.

(*e*) *Length.*—Hard temper bar, unless otherwise specified, and all rod and shapes shall be furnished in straight lengths. See Section 8(*c*) and Table XIII.

(*f*) *Straightness.*—See Section 9(*a*) and Table XV.

(*g*) *Edge Contours.*—See Section 10.

Sections 19, 20, 21, 22, 23, 24 and 25.—Delete.

Tentative Specifications for Seamless Brass Tubes (B 135 - 49 T):

New Section.—Add a new Section 2 to read as indicated above for new Section 2 in Specifications B 75, renumbering subsequent sections accordingly.

Sections 3, 5, 12, 13, 14 and 15.—Delete and renumber subsequent sections accordingly.

Section 16.—Revise to read as indicated above for Section 17 in Specifications B 75 except that in Paragraph (*d*) delete "Table XI."

Sections 17, 18, 19, 20, Explanatory Note and Appendix.—Delete.

Tentative Specifications for Phosphor Bronze Rods, Bars, and Shapes (B 139 - 50 T):

Section 1.—Revise to read as follows retaining the note: "This specification covers five alloys designated A, B1, B2, C and D, of phosphor bronze rod, bar, and shapes."

New Section.—Add a new Section 2 to read as indicated above for new Section 2 in Specifications B 21, renumbering subsequent sections accordingly.

Sections 3, 5, 8, 9 and 10.—Delete and renumber subsequent sections accordingly.

Section 11.—Revise to read as indicated above for Section 12 in Specifications B 21 except that "Tables I, VIII, X, and XIII" should read respectively "II, IX, XI, and XIV."

Section 12.—Delete and renumber subsequent sections accordingly.

Section 13.—Delete Paragraph (*c*).

Sections 14, 15, 16 and 17.—Delete.

Tentative Specifications for Aluminum Bronze Rods, Bars, and Shapes (B 150 - 49 T):

New Section.—Add a new Section 2 to read as indicated above for new Section 2 in Specifications B 21, renumbering subsequent sections accordingly.

Sections 3, 5, 9, 10 and 11.—Delete and renumber subsequent sections accordingly.

Section 12.—Revise to read as indicated above for Section 11 in Specifications B 139 except that in Paragraph (*b*) add Items (*2*) and (*3*) to read:

(*2*) *Rod and Bar, As-Extruded.*—See Table IV.

(*3*) *Round Rod, As-Hot-Rolled.*—See Table VI.

and in Paragraph (*e*) add Item (*3*) to read "(*3*) As-hot-rolled and as-extruded rod, bar, and shapes shall be commercially straight."

Section 13.—Delete and renumber subsequent sections accordingly.

Section 14.—Delete Paragraph (*c*).

Sections 15, 16, 17 and 18.—Delete.

Tentative Specifications for Copper-Nickel-Zinc Alloy Rods and Bars (B 151 - 50 T):

New Section.—Add a new Section 2 to read as indicated above for new Section 2 in Specifications B 21, renumbering subsequent sections accordingly.

Sections 2, 4, 6, 10, 11 and 12.—Delete and renumber subsequent sections accordingly.

Section 13.—Renumber as Section 8 and revise to read as follows:

8. *Dimensions and Tolerances* (*a*) The dimensions and tolerances for material covered by this specification shall be as prescribed in the Tentative Specification for General Requirements for Wrought Copper and Copper-Alloy Rod, Bar, and Shapes (ASTM Designation: B—),[2] with particular reference to Sections 8, 9, and 10 and the following tables of that specification.

(*b*) *Diameter or Distance Between Parallel Surfaces:*

(*1*) *Rod; Round, Hexagonal, Octagonal.*—See Table II.

(*2*) *Bar; Rectangular and Square.*—See Tables IX and XI.

(*c*) *Length.*—See Section 8(*c*), Tables XII and XIV.

(*d*) *Straightness.*—See Section 9(*a*) and Table XV.

(*e*) *Edge Contours.*—See Section 10.

Sections 14, 15, 16, 17 and 18.—Delete.

Tentative Specifications for Copper Sheet, Strip, and Plate (B 152 - 50 T):

New Section.—Add a new Section 2 to read as indicated above for new Section 2 in Specifications B 36, renumbering subsequent sections accordingly.

Sections 3 and 5.—Delete and renumber subsequent sections accordingly.

Table II.—Revise title to read "Tensile Strength Requirements and Approximate Rockwell Hardness Values for Rolled Tempers." Revise the third column heading to read: "Approximate Rockwell Hardness[a]," retaining the present footnote *a*.

Table III.—In the title after "Requirements" add "and Approximate Rockwell Hardness Values." Revise the third column heading to read "Approximate Rockwell Hardness[b]." Delete "Superficial 15-T Scale" and also reference to it in footnote *b*.

Section 11.—Revise to read as indicated above for Section 11 in Specifications B 36 except that "brass" should read "copper" and "Table I" should read "Table II."

Sections 15, 16, and 17.—Delete and renumber subsequent sections accordingly.

Section 18.—Revise to read as indicated above for Section 15 in Specifications B 36 except that Paragraph (*f*) should be added to read "(*f*) *Weight.*—Hot-rolled sheet and plate. See Table XIII."

Sections 19, 20, 21, 22, 23 and Appendix.—Delete.

Tentative Specifications for Phosphor Bronze Wire (B 159 - 49 T):

New Section.—Add a new Section 2 to read as follows:

2. *General Requirements.*—Material furnished ander this specification shall conform to the upplicable requirements of the current edition of the Tentative Specification for General Requirements for Wrought Copper Alloy Wire (ASTM Designation: B—).[2]

Sections 3, 5, 6, 9 and 10.—Delete and renumber subsequent sections accordingly.

Section 11.—Renumber as Section 7 and revise to read as follows:

7. *Dimensions and Tolerances* (*a*) The dimensions and tolerances for material covered by this specification shall be as prescribed in Section 8 and the following table of the Tentative Specification for General Requirements for Wrought Copper Alloy Wire (ASTM Designation: B—).[2]

(*b*) *Diameter.*—See Table II.

Sections 12, 13, 14, 15 and Explanatory Note.—Delete.

Tentative Specifications for Aluminum Bronze Sheet and Strip (B 169 - 50 T):

New Section.—Add a new Section 2 to read as indicated above for new Section 2 in Specifications B 36, renumbering subsequent sections accordingly.

Sections 3, 5, 8, 9 and 10.—Delete and renumber subsequent sections accordingly.

Section 11.—Revise to read as indicated above for Section 15 in Specifications B 36 except that "Table I" should read "Table II."

Sections 12, 13, 14, 15 and Appendix.—Delete.

Tentative Specifications for Seamless Copper Bus Pipes and Tubes (B 188 - 49 T):

New Section.—Add a new Section 2 to read as indicated above for new Section 2 in Specifications B 75, renumbering subsequent sections accordingly.

Sections 2, 4, 9, 10 and 11.—Delete and renumber subsequent sections accordingly.

Sections 12 and 13.—Combine, renumber as Section 8, and revise to read as follows:

8. *Dimensions, Weights, and Tolerances* (*a*) The dimensions, weights, and tolerances for material covered by this specification shall be as prescribed in the Tentative Specification for General Requirements for Wrought Seamless Copper and Copper-Alloy Pipe and Tube (ASTM Designation: B—),[2] with particular reference to Section 8 and the following tables of that specification.

(*b*) *Pipe Dimensions and Standard Weight.*—See Section 8(*b*), Table I.

(*c*) *Tube Dimensions and Weights.*—See Section 8(*b*), Tables II and III.

(*d*) *Weight Tolerances.*—See Section 8(*c*), Tables I and IV.

(*e*) *Wall Thickness Tolerances.*—See Section 8(*d*), Tables II and V.

(*f*) *Diameter Tolerances.*—See Section 8(*e*) Tables II and VIII.

(*g*) *Length and Length Tolerances.*—See Section 8(*g*) and (*h*), Tables IX and XI.

(*h*) *Roundness.*—See Section 8(*f*).

(*i*) *Squareness of Cut.*—See Section 8(*i*).

(*j*) *Straightness Tolerances.*—See Section 8(*j*).

(*k*) *Rectangular and Square Tubes.*—Tolerances for rectangular and square tubes shall be agreed upon by the purchaser and the manufacturer at the time of placing the order.

Sections 14, 15, 16 and 17.—Delete.

Tentative Specifications for Beryllium-Copper Alloy Plate, Sheet and Strip (B 194 - 50 T):

Section 1.—Revise beryllium content "2.05" to read "1.95."

TABLE I.—MECHANICAL TEST REQUIREMENTS FOR COLD-ROLLED PLATE, SHEET, AND STRIP AS SUPPLIED COMMERCIALLY.

Condition	Tensile Strength, psi	Rockwell Hardness[a]	
		B Scale	Superficial 30T Scale
A	60 000 to 78 000	45 to 78	46 to 67
¼ H	75 000 to 88 000	68 to 90	62 to 75
½ H	85 000 to 100 000	88 to 96	74 to 79
H	100 000 to 120 000	96 to 102	79 to 83

[a] The minimum thickness of material that may be tested in the case of the above Rockwell hardness scales is as follows:

	Thickness, in.
B Scale	0.032 and over
Superficial 30T Scale	0.010 and over

Table III.—Revise to read as shown in the accompanying Table II.

TABLE II.—MECHANICAL TEST REQUIREMENTS FOR COLD-ROLLED PLATE, SHEET, AND STRIP AFTER PRECIPITATION HARDENING.

Condition	Tensile Strength, psi	Rockwell Hardness[a]	
		C Scale	Superficial 30 N Scale
AT	160 000 to 190 000	34 to 40	55 to 61
¼ HT	170 000 to 200 000	36 to 42	57 to 63
½ HT	180 000 to 210 000	38 to 44	59 to 65
HT	185 000 to 215 000	39 to 45	60 to 66

[a] The minimum thickness of material that may be tested in the case of the above Rockwell hardness scales is as follows:

	Thickness, in.
C Scale	0.032 and over
Superficial 30 N Scale	0.010 and over

New Section.—Add a new Section 2 to read as indicated above for new Section 2 in Specifications B 36, renumbering subsequent sections accordingly.

Section 3.—Delete and renumber subsequent sections accordingly.

Section 4.—Revise beryllium content "1.90 to 2.15" to read "1.80 to 2.05."

Section 5.—Delete and renumber subsequent sections accordingly.

Table II.—Revise to read as shown in the accompanying Table I.

Sections 9, 10, 11 and 12.—Delete and renumber subsequent sections accordingly.

Section 13.—Revise to read as indicated above for Section 15 in Specifications B 36 except that "Table I" should read "Table II."

Sections 14, 15, 16, 17 and Appendix I.—Delete.

Tentative Specifications for Beryllium-Copper Alloy Rod and Bar (B 196 - 49 T):

Section 1.—Revise beryllium content "2.05" to read "1.95."

New Section.—Add a new Section 2 to read as indicated above for new Section 2 in Specifications B 21, renumbering subsequent sections accordingly.

Section 3.—Delete and renumber subsequent sections accordingly.

Section 4.—Revise beryllium content "1.90 to 2.15" to read "1.80 to 2.05."

Section 5.—Delete and renumber subsequent sections accordingly.

Table II.—Revise to read as shown in the accompanying Table III.

Table III.—Revise to read as shown in the accompanying Table IV.

Sections 9, 11, and 12.—Delete and renumber subsequent sections accordingly.

Section 13.—Revise to read as indicated above for Section 13 in Specifications B 151 except that in Paragraph (*b*) add Item (*2*) to read "(*2*) *Round Rod as Hot-Rolled.*—See Table VI," and in Paragraph (*d*) add Item (*2*) to read "(*2*) As-hot-rolled rod shall be commercially straight."

Sections 14, 15, 16, 17 and 18.—Delete.

Tentative Specifications for Beryllium-Copper Alloy Wire (B 197 - 50 T):

Section 1.—Revise beryllium content "2.05" to read "1.95."

TABLE III.—MECHANICAL TEST REQUIREMENTS FOR ROD AND BAR AS SUPPLIED COMMERCIALLY.

Condition	Diameter or Thickness, in.	Tensile Strength, psi	Rockwell Hardness[a] B Scale
A...	0.020 to 0.311, incl.........	60 000 to 85 000	...
	Over 0.311.....	60 000 to 85 000	45 to 85
H..	0.020 to 0.249, incl.........	95 000 to 130 000	...
	Over 0.249 to 0.375, incl...	95 000 to 130 000	92 to 103
	Over 0.375 to 1.000, incl...	90 000 to 120 000	91 to 102
	Over 1.000.....	85 000 to 115 000	88 to 101

[a] *Rockwell Hardness Testing:*
Location.—A flat surface may be prepared on round, hexagon or octagon rod which is parallel to the length and not more than one-quarter of the diameter or thickness from the original surface.
Preparation.—The flat may be prepared by either filing or grinding provided subsequent grinding yields a surface equivalent to or better than that produced by 150-grit.

TABLE IV.—MECHANICAL TEST REQUIREMENTS FOR ROD AND BAR AFTER PRECIPITATION HARDENING.

Condition	Diameter or Thickness, in.	Tensile Strength, psi	Rockwell Hardness[a] C Scale
AT...	0.020 to 0.249, incl........	160 000 to 190 000	...
	Over 0.249....	160 000 to 190 000	34 to 40
HT	0.020 to 0.249, incl........	185 000 to 215 000	...
	Over 0.249 to 0.375, incl..	185 000 to 215 000	39 to 45
	Over 0.375 to 1.000, incl..	180 000 to 210 000	38 to 44
	Over 1.000....	175 000 to 205 000	37 to 43

[a] *Rockwell Hardness Testing:*
Location.—A flat surface may be prepared on round, hexagon or octagon rod which is parallel to the length and not more than one-quarter of the diameter or thickness from the original surface.
Preparation.—The flat may be prepared by either filing or grinding provided subsequent grinding yields a surface equivalent to or better than that produced by 150-grit.

New Section.—Add a new Section 2 to read as indicated above for new Section 2 in Specifications B 159, renumbering subsequent sections accordingly.

Section 3.—Delete and renumber subsequent sections accordingly.

Section 4.—Revise beryllium content "1.90 to 2.15" to read "1.80 to 2.05."

Sections 5, 10, 11, 12 and 13.—Delete and renumber subsequent sections accordingly.

Section 14.—Revise to read as indicated above for Section 11 in Specifications B 159.

Sections 15, 16, 17 and Explanatory Note.—Delete.

Tentative Specifications for Copper-Nickel-Zinc Alloy Wire (B 206 – 50 T):

New Section.—Add a new Section 2 to read as indicated above for new Section 2 in Specifications B 159, renumbering subsequent sections accordingly.

Sections 3, 5, 11, 12 and 13.—Delete and renumber subsequent sections accordingly.

Section 14.—Revise to read as indicated above for Section 11 in Specifications B 159 except that Paragraphs (*c*), (*d*) and (*e*) should be inserted to read as follows:

(*c*) *Thickness.*—See Table IV.
(*d*) *Width.*—See Table VI.
(*e*) *Length (Flat Wire Only).*—See Table VII.

Sections 15, 16, 17, 18, 19 and Explanatory Note.—Delete.

WITHDRAWAL OF TENTATIVE

Tentative Specifications for Beryllium-Copper Alloy Strip, Special Grade (B 195 – 50 T):

The committee recommends that Tentative Specifications B 195 be withdrawn, since the material will be covered in the proposed revisions for Specifications B 194.

ADOPTION OF TENTATIVES AS STANDARD

The committee recommends that the following 3 tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Specifications for:

Cartridge Brass Cartridge Case Cups (B 129 - 49 T),

Gilding Metal Bullet Jacket Cups (B 131 - 49 T), and

Copper Bus Bars, Rods, and Shapes (B 187 - 49 T).

ADOPTION OF TENTATIVE REVISIONS OF STANDARDS AS STANDARD

The committee recommends the adoption as standard of the tentative revisions issued in 1949 in the following 5 standard specifications:

Standard Specifications for:

Copper Rods for Locomotive Staybolts (B 12 - 49),

Free-Cutting Brass Rod and Bar for Use in Screw Machines (B 16 - 49),

Copper-Silicon Alloy Rods, Bars, and Shapes (B 98 - 49),

Manganese Bronze Rods, Bars, and Shapes (B 138 - 49), and

Leaded Red Brass (Hardware Bronze) Rods, Bars, and Shapes (B 140 - 50).

REVISION OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends, for immediate adoption, revisions in 20 specifications as indicated below and accordingly asks for a nine-tenths affirmative vote at the Annual Meeting in order that these modifications may be referred to letter ballot of the Society.

Standard Specifications for Free-Cutting Brass Rod and Bar for Use in Screw Machines (B 16 - 49):

Title.—Add "Shapes" after "Bar."

New Section.—Add a new Section 2 to read as indicated above for new Section 2 in Specifications B 21, renumbering subsequent sections accordingly.

Section 3.—Delete and renumber subsequent sections accordingly.

Section 4.—Add a note under the chemical requirements to read as in Note 1 under Table I of Specifications B 21 - 50 T.[2]

Sections 5, 6, 9, 10 and 11.—Delete and renumber subsequent sections accordingly.

Section 12.—Revise to read as indicated above for Section 12 in Specifications B 21 except that Item (*2*) of Paragraph (*b*) and Item (*2*) of Paragraph (*e*) should be deleted.

Sections 13, 14, 15, 16 and 17.—Delete.

Standard Specifications for Bronze Castings for Turntables and Movable Bridges and for Bearing and Expansion Plates of Fixed Bridges (B 22 - 49):

Title.—Revise title to read "Bronze Castings for Turntables; Movable Bridges and Bridge Parts; Bearing and Expansion Plates of Fixed Bridges."

Section 1.—In Paragraph (*a*) revise to read "These specifications cover five alloys of bronze castings for turntables, movable bridges and bridge parts, and two alloys—."

In Paragraph (*b*) add the following:

Alloy E.—For bushings for bridge pins and similar applications where angular movement is slight and compressive stresses may attain 8,000 psi.

Table I.—Add requirements for alloy E as follows:

	Min.	Max.
Copper, per cent	**60.0**	**68.0**
Tin, per cent		0.20
Lead, per cent		**0.20**
Zinc, per cent	Remainder	
Iron, per cent	**2.0**	**4.0**
Aluminum, per cent	**3.0**	**7.5**
Manganese, per cent	**2.5**	**5.0**

Table II.—For alloy D revise physical requirements to read:

Tensile strength, min., psi	40 000
Yield strength, min., psi	18 000
Elongation in 2 in., min., per cent	20

Add requirements for alloy E to read as follows:

Deformation limit, min., psi	55 000
Permanent set in 1 in. under 100,000 psi., in	0.03 max.
Tensile strength, min., psi	110 000
Yield strength, min., psi	60 000
Elongation in 2 in., min., Per cent	12

Section 6.—Revise Paragraph (*c*) to read "Except for alloy E, the yield strength—," and add a sentence to read "Until such time as Committee B-5 shall have more information, the yield strength for alloy E shall be determined as the stress producing a permanent set of the material of 0.2 per cent as determined by the offset method."

Section 8.—At the end of Paragraph (*a*) revise to read "—except that Fig. 1 of B 208 shall be used for alloy E and is not applicable to the other alloys."

Section 9(a).—In the first sentence, revise to read: "—from each melt for alloy A, B, and C castings;".

Add a new second sentence to read: "For alloy E castings, one tension and one compression test shall be made for each four castings from the same melt and the same pattern."

Standard Specifications for Seamless Copper Pipe, Standard Sizes (B 42 – 49):

New Section.—Add a new Section 2 to read as indicated above for new Section 2 in Specifications B 75, renumbering subsequent sections accordingly.

Sections 3, 5, 11, 12, and 13.—Delete and renumber subsequent sections accordingly.

Sections 14 and 15.—Combine, renumber as Section 10, and revise to read as follows:

10. *Dimensions, Weights, and Tolerances* (*a*) The dimensions, weights, and tolerances for material covered by this specification shall be as prescribed in the Tentative Specification for General Requirements for Wrought Seamless Copper and Copper-Alloy Pipe and Tube (ASTM Designation: B—),[2] with particular reference to Section 8 and the following tables of that specification.

(*b*) *Standard Dimensions and Weights.*—See Section 8(*b*), Table I.

(*c*) *Weight Tolerances.*—See Section 8(*c*) Table IV.

(*d*) *Wall Thickness Tolerances.*—See Section 8(*d*), Table V.

(*e*) *Length and Length Tolerances.*—See Section 8(*g*) and (*h*).

(*f*) *Roundness.*—See Section 8(*f*).

(*g*) *Squareness of Cut.*—See Section 8(*i*).

Sections 16, 17, 18 and 19.—Delete.

Standard Specifications for Seamless Red Brass Pipe, Standard Sizes (B 43 – 49):

New Section.—Add a new Section 2 to read as indicated above for new Section 2 in Specifications B 75, renumbering subsequent sections accordingly.

Sections 3, 5, 11, 12 and 13.—Delete and renumber subsequent sections accordingly.

Sections 14 and 15.—Revise to read as indicated above for Sections 14 and 15 in Specifications B 42.

Sections 16, 17, 18, 19 and 20.—Delete.

Standard Specifications for Steam or Valve Bronze Castings (B 61 – 49), and Standard Specifications for Composition Brass or Ounce Metal Castings (B 62 – 49):

The proposed revisions of these two standards covers component castings of valves, flanges, and fittings comprise revisions of requirements for chemical analysis, hydrostatic test, number of tests, workmanship and finish, and certification, and the deletion of requirements for manufacture, inspection, and rejection. The revised specifications are appended hereto.[3]

Standard Specifications for Seamless Copper Tubing, Bright Annealed (B 68 – 49):

Section 1.—Revise in the second paragraph to read "Three types may be specified as indicated below. Unless otherwise specified, any one of these types may be supplied."

New Section.—Add a new Section 2 to read as indicated above for new Section 2 in Specifications B 75, renumbering subsequent sections accordingly.

Sections 1, 6 and 14.—Change the word "tubing" to read "tube."

Section 2.—Revise Item (*1*) to read "(*1*) Type of copper, if required (Sections 1, 4)."

Sections 3, 5, 11, 12 and 13.—Delete and renumber subsequent sections accordingly.

Section 14.—Revise to read as indicated above for Section 17 in Specifications B 75, except that paragraph (*g*) should be deleted

Sections 15, 16, 17 and 18.—Delete.

Standard Specifications for Seamless Copper Water Tube (B 88 – 50):

New Section.—Add a new Section 2 to read as indicated above for new Section 2 in Specifications B 75, renumbering subsequent sections accordingly.

Sections 7, 11, 12 and 13.—Delete and renumber subsequent sections accordingly.

Sections 14, 15, and 16.—Combine, renumber as Section 11, and revise to read as follows:

11. *Dimensions, Weights, and Tolerances* (*a*) The dimensions, weights, and tolerances for material covered by this specification shall be as prescribed in the Tentative Specification for General Requirements for Wrought Seamless Copper and Copper-Alloy Pipe and Tube (ASTM Designation: B—),[2] with particular reference to Section 8 and the following tables of that specification.

(*b*) *Standard Dimensions and Weights.*—See Section 8(*b*), Table II.

(*c*) *Weight Tolerances.*—See Section 8(*c*), Table II.

(*d*) *Wall Thickness Tolerances.*—See Section 8(*d*), Table II.

(*e*) *Diameter Tolerances.*—See Section 8(*e*), Table II.

(*f*) *Length and Length Tolerances.*—See Section 8(*g*) and (*h*), Tables IX and X.

(*g*) *Roundness.*—See Section 8(*f*).

(*h*) *Squareness of Cut.*—See Section 8(*i*).

(*i*) *Straightness Tolerances.*—See Section 8 (*j*).

Sections 17, 18, 19, 20 and 21.—Delete.

Standard Specifications for Copper-Silicon Alloy Plate and Sheet for Pressure Vessels (B 96 – 49):

Table I.—For alloy A revise the maximum silicon requirement of "3.5" to read "3.6" per cent.

Table III.—For alloy A-7 revise the silicon range of "2.85 to 3.35" to read "2.8 to 3.6" per cent, and the minimum manganese content of "0.85" to read "0.50" per cent.

Standard Specifications for Copper-Silicon Alloy Plate, Sheet, and Strip for General Purposes (B 97 – 50):

New Section.—Add a new Section 2 to read as indicated above for new Section 2 in Specifications B 36, renumbering subsequent sections accordingly.

Table I.—For alloy A revise the maximum silicon requirement of "3.5" to read "3.6" per cent.

Sections 3 and 5.—Delete and renumber subsequent sections accordingly.

Section 6.—Revise to read: "The tension test shall be the standard test for all tempers of copper-silicon alloy, and acceptance or rejection shall depend only on the tensile strength which shall conform to the requirements prescribed in Table II."

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[3] The revised specifications were accepted by the Society and appear in the 1951 Supplement to Book of Standards, Part 2.

Table II.—Revise the title to read "Tensile Strength Requirements, and Approximate Rockwell Hardness and Grain Size Values."

Revise the third column heading to read "Approximate Rockwell Hardness" and the fourth column heading to read "Approximate Grain Size, mm."

Section 7.—Revise to read as indicated above for Section 11 in Specifications B 36 except that "brass" should read "copper-silicon alloy" and "Table I or III" should read "Table II."

Sections 8, 9, and 10.—Delete and renumber subsequent sections accordingly.

Section 11.—Revise to read as indiated above for Section 15 in Specifications B 36 except that "Table I" should read "Table II" and Paragraph *(f)* should be added to read "*(f) Weight.*—Hot-rolled sheet and plate. See Table XIII."

Sections 12 and 13.—Delete and renumber subsequent sections accordingly.

Section 14.—Delete Paragraph *(b)*.

Sections 15 and 16.—Delete.

Table IX.—For alloy A-7 revise the silicon range of "2.85 to 3.35" to read "2.8 to 3.6" per cent and the minimum manganese content of "0.85" to read "0.50" per cent.

Appendix II.—Delete.

Standard Specifications for Copper-Silicon Alloy Rods, Bars, and Shapes (B 98-49):

New Section.—Add a new Section 2 to read as indicated above for new Section 2 in Specifications B 21, renumbering subsequent sections accordingly.

Sections 3 and 5.—Delete and renumber subsequent sections accordingly.

Table I.—For alloy A revise the maximum silicon requirement of "3.5" to read "3.6."

Sections 8, 9, and 10.—Delete and renumber subsequent sections accordingly.

Section 11.—Renumber as Section 7 and revise to read as follows:

7. *Dimensions and Tolerances (a)* The dimensions and tolerances for material covered by this specification shall be as prescribed in the Tentative Specification for General Requirements for Wrought Copper and Copper-Alloy Rod, Bar, and Shapes (ASTM Designation: B—),[2] with particular reference to Sections 8 and 9 and the following tables of that specification.

(b) Diameter or Distance Between Parallel Surfaces:

(1) Rod; Round, Hexagonal, Octagonal.—For Alloys A, C, and D see Table II and for alloy B see Table I.

(2) Rod; Round, As-Hot-Rolled.—See Table VI.

(3) Bar; Rectangular and Square.—For alloys A, C, and D see Tables IX and XI and for alloy B see Tables VIII and X.

(c) Shapes.—The dimensional tolerances for shape shall be as agreed upon by the manufacturer and the purchaser and shall be specified in the order.

(d) Length.—See Section 8*(c)* and Tables XII and XIV.

(e) Straightness:

(1) Rod, Bar, and Shapes.—See Section 9*(a)* and Table XV.

(2) As-hot-rolled and as-extruded rod, bar, and shape shall be commercially straight.

Sections 12, 13, 14, 15 and 16 and Explanatory Note.—Delete.

Appendix.—For alloy A-7 revise the silicon range of "2.85 to 3.35" to read "2.8 to "3.6 per cent and the minimum manganese content of "0.85" to read "0.50" per cent.

Standard Specifications for Copper-Silicon Alloy Wire for General Purposes (B 99-49):

New Section.—Add a new Section 2 to read as indicated above for new Section 2 in Specifications B 159, renumbering subsequent sections accordingly.

Section 3.—Delete and renumber subsequent sections accordingly.

Section 4.—For alloy A revise the maximum silicon requirement of "3.5" to read "3.6" per cent.

Sections 5, 8, 9 and 10.—Delete and renumber subsequent sections accordingly.

Section 11.—Revise to read as indicated above for Section 11 in Specifications B 159 except that Paragraph (*b*) should read "(*b*) *Diameter.*—For alloy B see Table I, and for alloys A and C see Table II."

Sections 12, 13, 14, 15 and Explanatory Note.—Delete.

Appendix.—For alloy A-7 revise the silicon range of "2.85 to 3.35" to read "2.8 to 3.6" per cent, and the minimum manganese content of "0.85" to read "0.50" per cent.

Standard Specifications for Phosphor Bronze Plate, Sheet, and Strip (B 103 – 50):

New Section.—Add a new Section 2 to read as indicated above for new Section 2 in Specifications B 36, renumbering subsequent sections accordingly.

Sections 3 and 5.—Delete and renumber subsequent sections accordingly.

Section 6.—Renumber as Section 5 and revise the last sentence to read "Rockwell hardness and tensile strength values shown in Table II for the various alloys in the annealed condition should be considered as approximate only (see Section 8)."

Section 8.—Delete Paragraph (*b*).

Section 9.—Renumber as Section 8 and revise to read "Since Rockwell hardness tests offer a quick and convenient method of checking phosphor bronze of any temper for general conformity to the requirements for tensile strength or grain size, the approximate Rockwell hardness values for each temper are given in Table II for general information and assistance in testing."

Table II.—Revise the title to read: "Tensile Strength Requirements and Approximate Rockwell Hardness Values."

Revise the fourth column heading to read "Approximate Rockwell Hardness" and throughout the table delete "Phosphor Bronze Plate, Sheet, and Strip."

Sections 10, 11, and 12.—Delete and renumber subsequent sections accordingly.

Section 13.—Renumber as Section 9 and revise to read as indicated above for Section 15 in Specifications B 36 except that "Table I" should read "Table II."

Sections 14, 15, 16, 17 and Appendix.—Delete.

Standard Specifications for Copper and Copper-Alloy Seamless Condenser Tubes and Ferrule Stock (B 111 – 50):

Section 4.—Revise to read as follows:

4. *Temper* (*a*) Muntz metal, admiralty metal, red brass, aluminum brass, aluminum bronze, 70-30 copper-nickel and 80-20 copper-nickel tubes shall normally be furnished in annealed temper.

(*b*) Copper or arsenical copper tubes shall be supplied in any one of the following tempers, unless otherwise specified:

Light-drawn temper
Hard-drawn temper
Hard-drawn temper, end-annealed

(*c*) 90-10 copper-nickel tubes may be supplied in either light-drawn temper or annealed temper.

(*d*) Tubes supplied in light-drawn temper shall pass the expanding test specified in Section 8(*a*).

(*e*) Tubes for ferrule stock shall be annealed sufficiently to be fully recrystallized, but are not subject to the expanding test specified in Section 8.

Section 8.—In the first sentence of Paragraph (*a*) revise to read "The annealed or light-drawn temper tubes selected for test shall be capable of withstanding the following tests without showing—."

Standard Specifications for Copper and Copper-Base Alloy Forging Rods, Bars, and Shapes (B 124 – 49):

New Section.—Add a new Section 2 to read as indicated above for new Section 2 in Specifications B 21, renumbering subsequent sections accordingly.

Sections 3, 5, 6 and 7.—Delete and renumber subsequent sections accordingly.

Section 8.—Renumber as Section 5 and revise to read as follows:

[5. *Dimensions and Tolerances* (*a*) The dimensions and tolerances for material covered by this specification shall be as prescribed in the Tentative Specification for Wrought Copper and Copper-Alloy Rod, Bar, and Shapes (ASTM Designation: B—),[2] with particular reference to Sections 8 and 9 and the following tables of that specification.

(*b*) *Diameter or Distance Between Parallel Surfaces:*

(*1*) *Hot-Rolled Round Rod.*—See Table VI.

(*2*) *As-Extruded Rod and Bar Including Round, Square, Rectangular, Hexagonal and Octagonal.*—For alloys 1, 2, 3, 4, 5, 11*a* and 11*b* see Table IV and for alloys 6, 7, 8, 9, 10 and 12 see table V.

(*c*) *Shapes.*—The dimensional tolerances for shape shall be as agreed upon by the manufacturer and the purchaser and shall be specified in the order.

(*d*) *Length.*—See Section 8(*c*) (*4*).

(*e*) *Straightness.*—The material shall be commercially straight.

Sections 9, 10, and 11.—Delete.

Standard Specifications for Brass Wire (B 134 – 50):

New Section.—Add a new Section 2 to read as indicated above for new Section 2 in Specifications B 159, renumbering subsequent sections accordingly.

Sections 3, 5, 10, 11 and 12.—Delete and renumber subsequent sections accordingly.

Section 13.—Renumber as Section 9 and revise to read as follows:

9. *Dimensions and Tolerances* (*a*) The dimensions and tolerances for material covered by this specification shall be as prescribed in the Tentative Specification for General Requirements for Wrought Copper Alloy Wire (ASTM Designation: B—),[2] with particular reference to Section 8 and the following tables of that specification.

(*b*) *Diameter.*—See Table I.

(*c*) *Thickness.*—See Table III.

(*d*) *Width.*—See Table V.

(*e*) *Length* (*for Flat Wire Only*).—See Table VII.

Sections 14, 15, 16, 17, 18 and Explanatory Note.—Delete.

Standard Specifications for Manganese Bronze Rods, Bars, and Shapes (B 138 – 49):

Section 1.—Revise to read: "This specification covers manganese bronze rod, bar, and shapes having the following compositions:—"

New Section.—Add a new Section 2 to read as indicated above for new Section 2 in Specifications B 21, renumbering subsequent sections accordingly.

Sections 3 and 5.—Delete and renumber subsequent sections accordingly.

Table II.—For alloy A revise tensile requirements for hard temper rods and bars to read as shown in the accompanying Table V.

TABLE V

Form	Temper	Diameter or Distance Between Parallel Surfaces, in.	Tensile Strength, min., psi.	Yield Strength, min., psi.	Elongation in 4 × Diameter, per cent
Rods...	Hard	1.0 and under...	80 000	56 000	8
		Over 1.0 to 1.5, incl.	76 000	52 000	10
		Over 1.5 to 2.5, incl.	73 000	48 000	12
		Over 2.5........	68 000	45 000	16
Bars...	Hard	1.0 and under...	76 000	52 000	8
		Over 1.0 to 2.5, incl.	72 000	47 000	12
		Over 2.5........	68 000	45 000	16

Sections 9, 10, and 11.—Delete and renumber subsequent sections accordingly.

Section 12.—Revise to read as indicated above for Section 12 in Specifications B 150 except that Item (*2*) of Paragraph (*b*) should read "(*2*) *Rod and Bar, As-Extruded.*—For alloy A see Table IV, and for alloy B see Table V."

Paragraph (*d*) should read "(*d*) *Length of Rod, Bar, and Shapes.*—See Section

8(*c*). For alloy A see Table XII and for alloy B see Table XIII."

Section 13.—Delete and renumber subsequent sections accordingly.

Section 14.—Delete Paragraph (*c*).

Sections 15, 16, 17 and 18.—Delete.

Standard Specifications for Leaded Red Brass (Hardware Bronze) Rods, Bars, and Shapes (B 140 – 50):

New Section.—Add a new Section 2 to read as indicated above for new Section 2 in Specifications B 21, renumbering subsequent sections accordingly.

Sections 3, 5, 8, 9 and 10.—Delete and renumber subsequent sections accordingly.

Section 11.—Revise to read as indicated above for Section 12 in Specifications B 21 except that Item (*2*) of Paragraph (*b*) and Item (*2*) of Paragraph (*e*) should be deleted.

Sections 12, 13, 14, 15 and 16.—Delete.

Standard Method of Mercurous Nitrate Test for Copper and Copper Alloys (B 154 – 50):

The proposed revision of this method includes requirements as to scope, mercurous nitrate solution, test specimen and procedure, and the deletion of requirements as to apparatus and report The revised method is appended hereto.[4]

Standard Specifications for Copper-Alloy Condenser Tube Plates (B 171 – 49):

Section 1.—In the second line revise "five" to read "six."

Table I.—Revise the last column heading to read "Aluminum Bronze, Alloy E," and add "Aluminum Bronze, Alloy D" with the following composition:

Copper, per cent	88.0 to 92.5
Iron, per cent	1.5 to 3.5
Aluminum, per cent	6.0 to 8.0
Copper plus sum of named elements, min., per cent	99.5

Section 6.—Revise "Aluminum bronze" to read "Aluminum bronze, Alloy E" and add "Aluminum bronze, Alloy D" with the following properties:

Tensile Strength min. psi	70 000
Yield Strength, min. psi	30 000
Elongation in 2 in., min., per cent	35.0

Section 7.—In the second sentence revise "Aluminum bronze" to read "Aluminum bronze, alloy E."

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[4] The revised method was accepted by the Society and appears in the 1951 Supplement to Book of ASTM Standards, Part 2.

REPORT OF COMMITTEE B-6

ON

DIE-CAST METALS AND ALLOYS*

Committee B-6 on Die-Cast Metals and Alloys held one meeting in Atlantic City, N. J., on June 28, 1950.

There is a balance of $2614.81 in the funds of Committee B-6, as of December 31, 1950.

At the present time, the committee consists of 92 members, of whom 43 are classified as producers, 24 as consumers, and 25 as general interest members.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends that the Tentative Specifications for Magnesium-Base Alloy Die Castings (B 94 - 49 T) and the Tentative Specifications for Zinc-Base Alloys in Ingot Form for Die Castings (B 240 - 49 T) be continued as tentative without revision.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II on Zinc-Base Die-Casting Alloys (A. E. Weiss, chairman) reported the appointment of a Task Group to set limits for impurities generally found in die castings but which are not at present covered in the Specifications for Zinc-Base Alloy Die Castings (B 86 - 48).

Subcommittee IX on Die-Casting Processes (A. E. Martin, chairman) reported the completion of the design of a test casting die for hot or cold chamber casting of zinc alloys.

The work fostered by Committee B-6 on exposure testing and stability of die castings and subsequent work by the industry had established the fact that stable die-casting alloys were available. It was felt, however, that much less was known about the die-casting process. The quality of the casting is as dependent upon the process as upon the alloy composition. For example, there was need for knowing whether high or low pressures would produce the sounder casting. In general, it was felt that if the process were better understood and controlled, wider application of die castings would ensue. Following a series of discussions among Gordon C. Curry, David Basch, and J. R. Townsend, invitations were directed to representatives of a number of die-casting companies to take part in a discussion of this problem. This meeting, under ASTM sponsorship, was held on January 15, 1946. As a result of the interest shown, several subsequent meetings were held, and on June 19, 1947, Subcommittee IX of ASTM Committee B-6 on Die Casting Processes was organized, with A. E. Martin as chairman and with a membership which included almost all leading producers and users of die castings and of die-casting alloys.

Shortly after the organization of this group, an Advisory Group was created, together with task groups specifically charged with the investigation of certain factors pertinent to the die-casting process, including pressure, temperature, plunger speed, melting practices, die gating and venting, and die lubrication. Among these, Task Group E was requested by the Advisory Group to conduct an investigation of the effect of pressure on the quality of aluminum-base alloy castings. It was originally suggested that the pressure range between 500 and 100,000 psi should be explored, employing two gate thicknesses, having a thickness ratio of approximately 3 to 1. This investigation, the details of the work, and the conclusions drawn from the data obtained, are covered in a paper on "Aluminum Die Castings—The Effect of Process Variables on Their Properties" by W. Babington and D. H. Kleppinger. This paper is appended to this report.

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* Presented at the Fifty-fourth Annual Meeting of the Society, June 18-22, 1951.

CODE SYSTEM FOR ALLOYS

The Task Group appointed to study the negative votes cast on the adoption of the Frankford Arsenal code identification system for alloys and to devise a satisfactory compromise, recommended that:

1. The code system be adopted without change for the aluminum alloys;
2. The code be resubmitted to letter ballot but this time members are to vote on the adoption of the system for the alloys in each of the six specifications; and
3. If the code is adopted for the zinc alloys, the old and the new system numbers shall be carried concurrently in the specifications.

As recommended, the code was resubmitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.

This report has been submitted to letter ballot of the committee, which consists of 92 members; 85 members returned their ballots, of whom 76 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

J. R. TOWNSEND,
Chairman.

G. L. WERLEY,
Secretary.

APPENDIX

ALUMINUM DIE CASTINGS—THE EFFECT OF PROCESS VARIABLES ON THEIR PROPERTIES*

By W. Babington[1] and D. H. Kleppinger[2]

For many years there has been much speculation relative to the effect of certain variables inherent in the die-casting process upon the quality of die castings produced. Prior to World War II, a substantial number of the aluminum-base die castings produced in this country were made in the so-called "goose-neck" die-casting machine, employing relatively low injection pressures. Under the impetus of World War II, the demand was created for aluminum-base alloy die castings which would withstand higher stresses than those previously produced. As a result, the "goose-neck" type machines were largely supplanted by the "cold-chamber" machines, employing injection pressures considerably higher than those previously used.

Among the advantages claimed for castings produced under higher pressures in the cold-chamber machines were lower iron content, improved density, and better mechanical properties. As a result, the trend in the manufacture of die-casting machines has been in the direction of increasing injection pressures. A survey of the literature on this subject disclosed little information as to whether the improvements observed were due to high injection velocity or to high pressure on the solidifying metal.

The organization of Subcommittee IX on Die-Casting Processes, of ASTM Committee B-6 on Die-Cast Metals and Alloys, was an outgrowth of the consensus of leading die casters and users of die castings that certain advantages would accrue from an exploration of some of the fundamentals of the die-casting process. Accordingly, within this subcommittee, a number of task groups were set up for the purpose of investigating the various die-casting factors believed to affect the quality of die castings.

Experimental Work

Test Casting Die:

The first work undertaken by this subcommittee was the design of a test casting die (Fig. 1) which would incorporate many of the features of commercial castings, such as thick and thin wall sections, relatively large flat areas, abrupt changes in sections, and cored holes. The design also included provisions for a test area in the casting from which suitable tension and impact test bars could be produced for mechanical property evaluation. The casting produced in this die is described in a paper by E. Jacobi.[3] This die was intended to serve as a tool for later studies of the process variables. At the suggestion of the Advisory Group of Subcommittee IX, Task Group E was created for the purpose of studying the effect of pressure on the quality of aluminum die castings. A specific suggestion was made by the Advisory Group that this study be made using casting pressures in the range of 500 to 100,000 psi, employing two widely varying gate thicknesses of approximately the ratio of 3 to 1. The work covered in this paper is the outgrowth of this recommendation.

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* Presented at the Fifth-fourth Annual Meeting of the Society, June 18–22, 1951.

[1] Member of Technical Staff, Bell Telephone Laboratories, Inc., Murray Hill, N. J.

[2] Metallurgist, Frankford Arsenal, Phila., Pa.; The opinions expressed in this report are those of the author and do not necessarily represent the views of the Ordnance Corps nor the Department of the Army.

[3] E. Jacobi, "Flow Calculations for Die Casting Applied to the ASTM Committee B-6 Test Casting Die," ASTM Bulletin, No. 166, May, 1950, p. 65 (TP127).

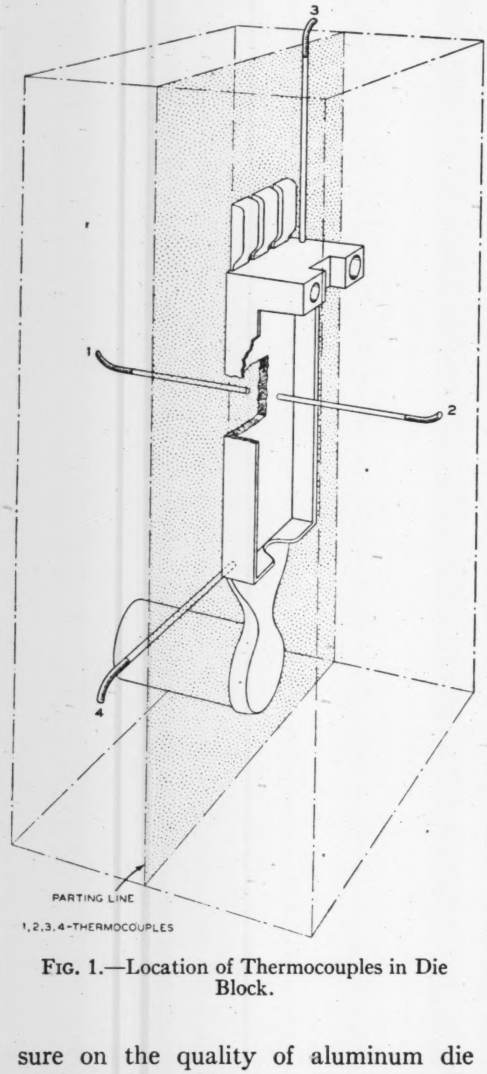


FIG. 1.—Location of Thermocouples in Die Block.

The instrumentation was developed with the assistance of various members of the subcommittee as reported elsewhere in this paper.

The castings were produced, with the aid of this instrumentation, in the Die Casting Development Laboratory of the Bell Telephone Laboratories, Inc., Murray Hill, N. J. The radiographs, preparation of tension specimens and the mechanical tests were done at the Pitman-Dunn Laboratory of the Frankford Arsenal, Philadelphia, Pa. The visual inspection, radiographic rating and weight determinations were conducted jointly. The test data were reduced to curves at the Bell Telephone Laboratories and the conclusions arrived at jointly.

Test Program:

The test program consisted of producing castings in the ASTM test die (see Fig. 1), within the suggested range of pressures, using gate thicknesses of 0.030 in. and 0.090 in., while holding all temperature and speed factors as constant as practical. The castings so made were to be subjected to visual, radiographic, and mechanical tests for the evaluation of relative quality.

Pressure Measurement:

In the initial discussion of this program, the term "pressure" was interpreted as the calculated plunger pressure normally used in the die-casting industry. This value is the product of the hydraulic line pressure and the ratio of the areas of the hydraulic cylinder and plunger. It is at once evident that the use of calculated pressure values presents considerable complication. Such pressure values do not take into account pressure losses due to friction in the hydraulic system and losses which will occur in the hydraulic cylinder and the plunger. It was also realized that these values will vary considerably between two die-casting machines and might conceivably show substantial variations from time to time on the same die-casting machine, depending upon the operating conditions. In view of this, it was felt that the pressure values employed in this study should be actual pressures determined on the molten metal system. Since no known method of making such pressure measurements was available, it was agreed that the preliminary phase of this investigation should be devoted to the development of a suitable means of measuring the metal pressure within the die-casting cavity. A number of existing pressure-measuring systems were investigated, including the piezoelectric gages, copper pressure cylinders, and pressure-sensitive diaphragms. None of these systems, however, was considered satisfactory under the pressure and temperature conditions encountered in the die-casting process.

After considerable experimentation, the SR-4 resistance type strain gage was adapted to this application. The ASTM test-casting die is so constructed that the metal being injected into the die impinges directly upon the ends of two $\frac{3}{8}$-in. ejector pins located at the "biscuit" or sprue section of the die cavity. It was felt that at this location the metal pressure would be maximum and positive. Therefore, it was decided to measure the metal pressure exerted on these two ejector pins. Since it was impracticable to apply SR-4 gages directly to the ejector pins, the gages were installed on heat-treated beryllium-copper cylinders which in turn were mounted behind the ejector pins and restrained the movement of these pins in the ejector plate. In this manner, any pressure applied to the ends of the ejector pins was transmitted directly to the beryllium-copper pressure cylinders. Beryllium copper was used for this purpose, rather than steel, because its relatively low modulus of elasticity and high elastic strength afforded 60 per cent greater pressure sensitivity.

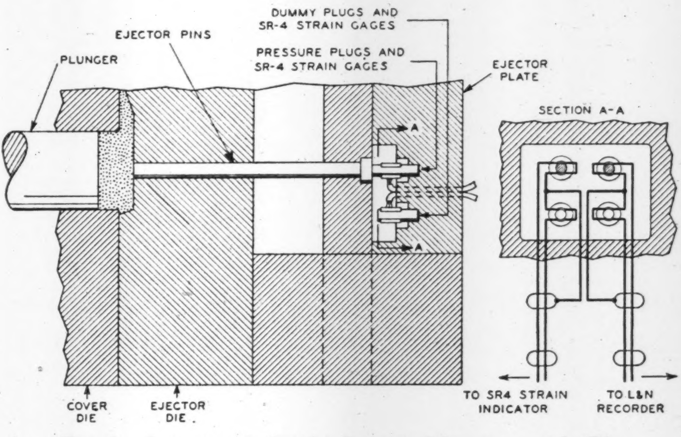
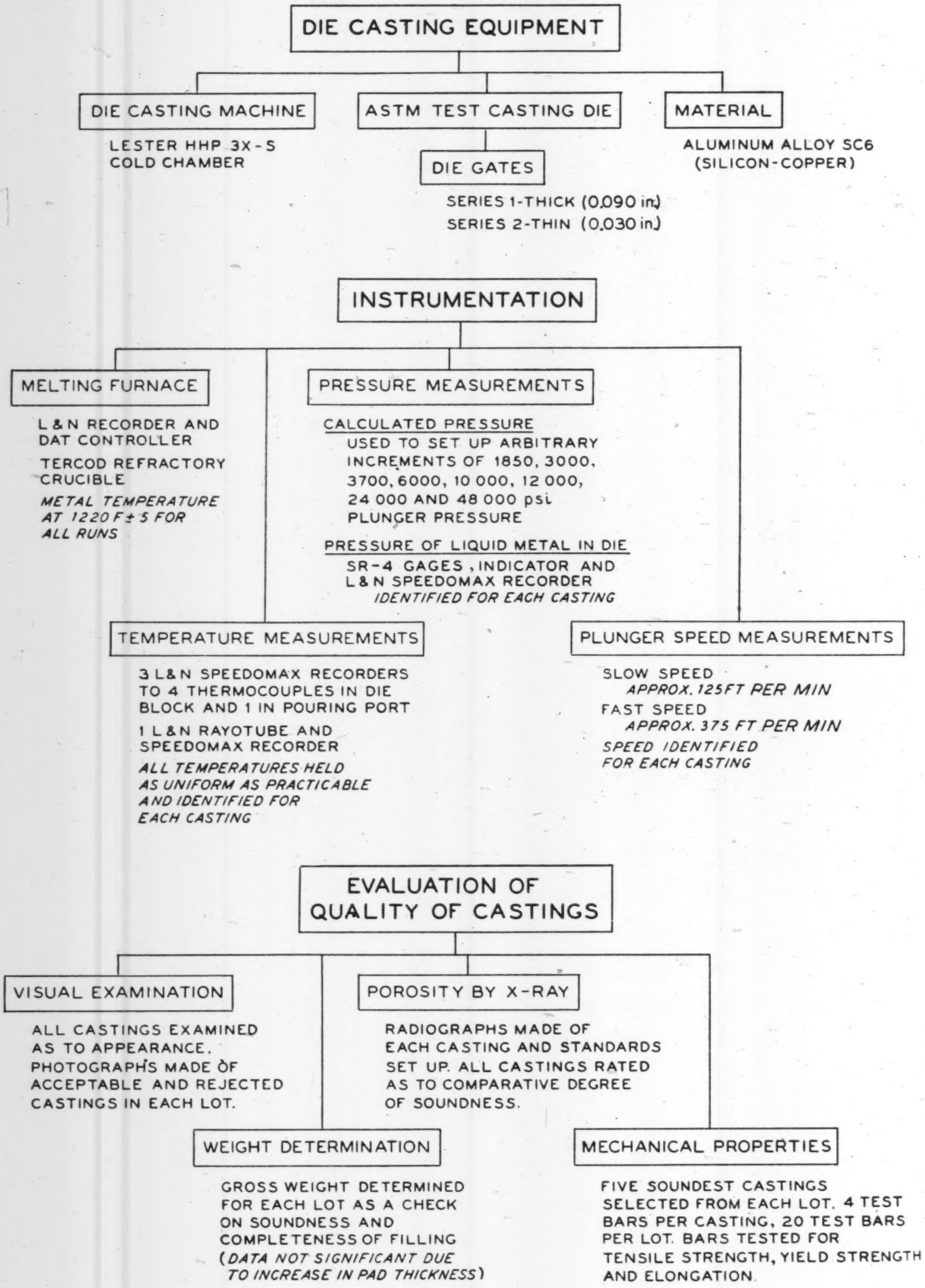


FIG. 2.—Instrumentation for Pressure Measurement in Die.

Figure 2 shows the manner in which the beryllium-copper pressure-measuring cylinders (1 in. long by $\frac{5}{16}$ in. in diameter) were installed in the test-casting die. The installation consists of four beryllium-copper cylinders, two of which are active pressure-measuring cylinders,


DIE CASTING EQUIPMENT
DIE CASTING MACHINE
ASTM TEST CASTING DIE
MATERIAL
DIE GATES
INSTRUMENTATION
MELTING FURNACE
PRESSURE MEASUREMENTS
TEMPERATURE MEASUREMENTS
PLUNGER SPEED MEASUREMENTS
EVALUATION OF QUALITY OF CASTINGS
VISUAL EXAMINATION
POROSITY BY X-RAY
WEIGHT DETERMINATION
MECHANICAL PROPERTIES

LESTER HHP 3X-S
COLD CHAMBER

SERIES 1-THICK (0.090 in.)
SERIES 2-THIN (0.030 in.)

ALUMINUM ALLOY SC6
(SILICON-COPPER)

L & N RECORDER AND DAT CONTROLLER

TERCOD REFRACTORY CRUCIBLE

METAL TEMPERATURE AT 1220 F ± 5 FOR ALL RUNS

CALCULATED PRESSURE
USED TO SET UP ARBITRARY INCREMENTS OF 1850, 3000, 3700, 6000, 10 000, 12 000, 24 000 AND 48 000 psi PLUNGER PRESSURE

PRESSURE OF LIQUID METAL IN DIE
SR-4 GAGES, INDICATOR AND L & N SPEEDOMAX RECORDER
IDENTIFIED FOR EACH CASTING

3 L & N SPEEDOMAX RECORDERS TO 4 THERMOCOUPLES IN DIE BLOCK AND 1 IN POURING PORT

1 L & N RAYOTUBE AND SPEEDOMAX RECORDER

ALL TEMPERATURES HELD AS UNIFORM AS PRACTICABLE AND IDENTIFIED FOR EACH CASTING

SLOW SPEED
APPROX. 125 FT PER MIN
FAST SPEED
APPROX. 375 FT PER MIN
SPEED IDENTIFIED FOR EACH CASTING

ALL CASTINGS EXAMINED AS TO APPEARANCE. PHOTOGRAPHS MADE OF ACCEPTABLE AND REJECTED CASTINGS IN EACH LOT.

RADIOGRAPHS MADE OF EACH CASTING AND STANDARDS SET UP. ALL CASTINGS RATED AS TO COMPARATIVE DEGREE OF SOUNDNESS.

GROSS WEIGHT DETERMINED FOR EACH LOT AS A CHECK ON SOUNDNESS AND COMPLETENESS OF FILLING (*DATA NOT SIGNIFICANT DUE TO INCREASE IN PAD THICKNESS*)

FIVE SOUNDEST CASTINGS SELECTED FROM EACH LOT. 4 TEST BARS PER CASTING, 20 TEST BARS PER LOT. BARS TESTED FOR TENSILE STRENGTH, YIELD STRENGTH AND ELONGATION.

FIG. 3.—Chart for Investigation of Die Casting Process Variables.

mounted directly behind the ejector pins, and two are dummy cylinders located adjacent to afford temperature compensation. SR-4 gages were mounted on diametrically opposite sides of each cylinder, thus providing compensation for bending strain as well as temperature effects. As shown, the gages on each pair of cylinders (consisting of one active and one dummy) are wired in series and leads are provided to the strain-measuring instruments. In this manner an independent pressure-measuring system is established for each of the two ejector pins. These systems were then calibrated for strain *versus* compression in the die-casting machine and at casting temperatures using a Morehouse proving ring which had been certified by the Bureau of Standards. Subsequent calibrations were made periodically throughout this investigation. No appreciable variation in calibration was observed at any time.

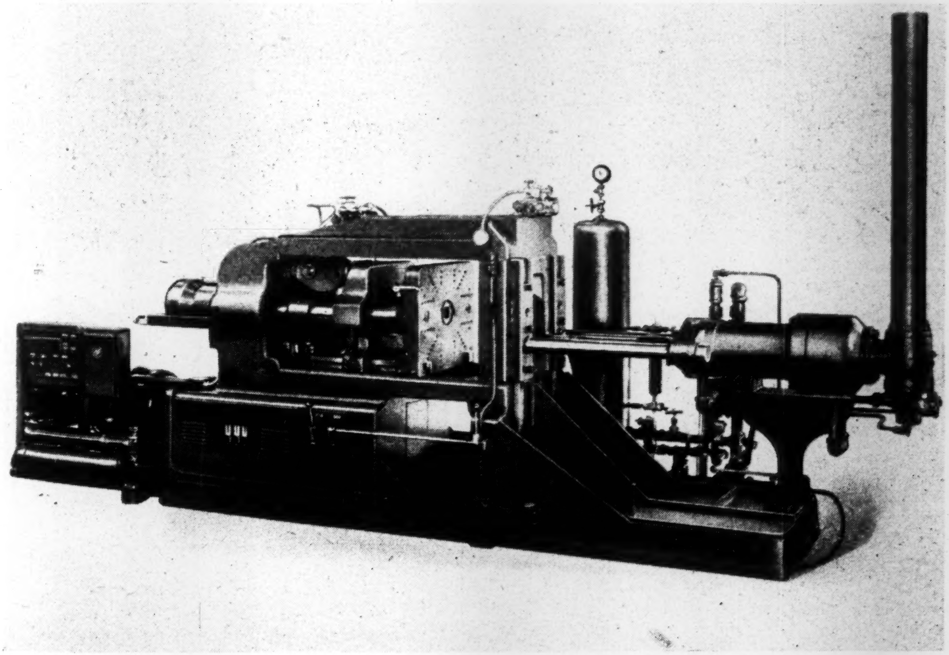


FIG. 4.—Type of Die Casting Machine Used in Investigation.

By this calibration, the strains measured by the SR-4 indicators in terms of microinches, were translated into pressure in pounds per square inch.

In the initial stages of the study, the strains were measured on a standard Baldwin SR-4 indicator and also on a Curtis string galvanometer rapid record oscillograph[4]. It was determined that accurate strain readings could be obtained on the SR-4 indicator and in agreement with the Curtis oscillograph results. Since the SR-4 indicator could be read directly, it was employed for the bulk of this work. During this phase of the investigation, cooperative work was being carried on by the Leeds & Northrup Co., in the development of a direct-reading electronic recorder capable of recording the strains produced on the SR-4 gages. As a result of this work, a recorder was perfected and placed at the disposal of the Bell Telephone Laboratories for use in this investigation. Experimentation indicated that an extremely close check of strain readings was obtained between the SR-4 strain indicator and the electronic recorder.

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[4] A. M. Curtis and I. E. Cole, "A Rapid Record Oscillograph," *Electronics*, Vol. III, August, 1931, pp. 70–71.

A series of independent casting runs within the range of 4000 to 50,000 psi was conducted on the ASTM casting die with a gate thickness of 0.060 in., for the purpose of determining the adequacy of this pressure-measuring device. This work showed that the instrumentation was adequate for conducting the proposed pressure study and that the measured metal pressures were approximately 20 per cent lower than the calculated pressures within the range studied. It should be pointed out that while it had originally been planned to conduct pressure studies up to 100,000 psi, the limitations of the available die-casting equipment made it impracticable to operate above 50,000 psi.

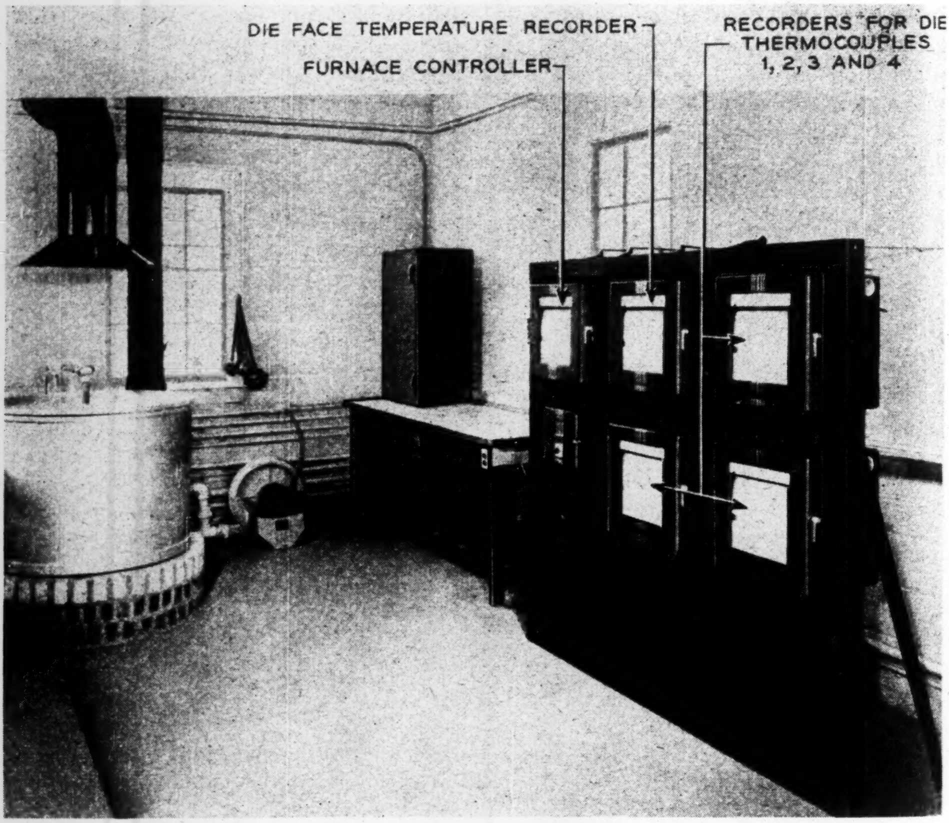
DIE FACE TEMPERATURE RECORDER
FURNACE CONTROLLER
RECORDERS FOR DIE THERMOCOUPLES 1, 2, 3 AND 4

FIG. 5.—Temperature Measurement Instrumentation.

DIE-CASTING OPERATION

Upon completion of the experimental work required to develop a suitable means of measuring metal pressures within the die cavity, work was resumed to determine the effect of metal pressures on the quality of die castings produced.

Materiel:

Figure 3 is a flow sheet indicating the various phases covered in this investigation. The first section of this flow sheet covers the materiel employed in making this study and includes:

1. A standard production type die-casting unit manufactured by the Lester-Phoenix Co. of Cleveland, Ohio, under their designation HHP-3X-S as shown in Fig. 4. This is a cold-chamber type machine with a nominal die-locking pressure of 500 tons, equipped with a prefill system and a nitrogen accumulator.

2. The ASTM test-casting die, as used for these studies, contained a vent 0.010 in. deep and 3 in. wide, running from the top of the casting cavity to the upper edge of the die block. When additional venting was required under certain casting conditions, 0.010-in. shims were inserted at the parting line on the upper liner pins.

3. The metal employed for all castings was a commercial shipment of the aluminum-silicon-copper alloy SC6 conforming to ASTM Specification B85.[5] The analysis of this alloy was as follows:

Elements	Per cent
Copper	3.42
Silicon	8.55
Manganese	0.12
Magnesium	0.02
Iron	0.42
Nickel	<0.01
Tin	<0.01
Zinc	<0.01

The instrumentation employed for the measurement of temperature, metal pressure, and plunger speed was as accurate as practical. Every effort was made to hold those casting conditions not under investigation as constant as possible and to measure the variables being studied with the highest degree of accuracy compatible with efficient operation of the die-casting unit.

The furnace for melting and holding the alloy for casting purposes was gas-fired, equipped with a "Tercod" refractory crucible. This type of crucible was used to minimize iron pick-up. Metal temperature control was achieved by a Leeds & Northrup "Duration-Adjusting Type" controller, which maintained the metal temperature at 1220 ± 5F throughout all the casting runs. It had been determined with the test casting die that at this temperature the SC6 alloy was readily castable.

Temperature Measurements:

Temperature measurements within the die block were made by means of four iron-constantan thermocouples located within the body of the die block, as shown in Fig. 1. These thermocouples were of the bayonet type and were silver tipped to afford rapid thermal conduction. A similar thermocouple was also located in the sleeve at a point immediately below the pouring port. These five thermocouples were connected to three Speedomax recorders with reversing switches, permitting the simultaneous measurement of temperature at any three desired locations. These temperatures were recorded and identified with each casting produced throughout all

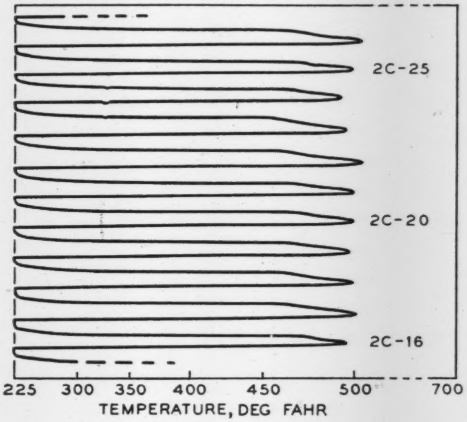

FIG. 6.—Typical Chart of Die Face Temperature.

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[5] Tentative Specifications for Aluminum-Base Alloy Die Castings (B 85 - 50 T), 1950 Supplement to Book of ASTM Standards, Part 2, p. 193.

the runs. Die-face temperatures were measured by means of a Rayotube black-body reflector and were recorded on a Speedomax recorder upon each opening of the die in the casting cycle. Figure 5 indicates the instrumentation employed for temperature recording. Typical temperature measurements obtained from the die face are shown in Fig. 6.

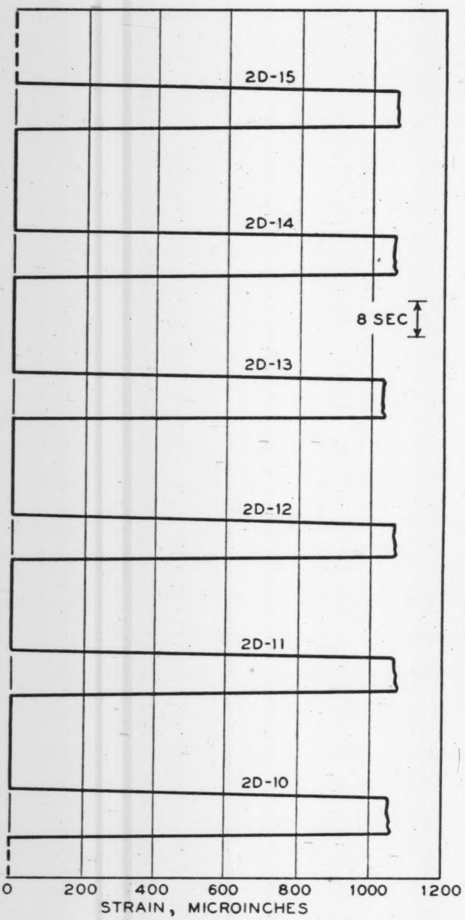


FIG. 7.—Typical Chart of Strain Measurements for Metal Pressure.

Metal Pressure:

The method of measuring metal pressure within the die cavity has been previously described. Calculated pressures were used as guides to set up the die-casting equipment for each successive casting condition. For each casting produced, metal pressure measurements were made simultaneously from one ejector pin on an SR-4 strain indicator and from the other pin, on a Speedomax recorder. These strain values were later converted to pressure readings by means of a calibration chart. Figure 7 indicates a typical series of strain measurements in microinches taken from the recorder.

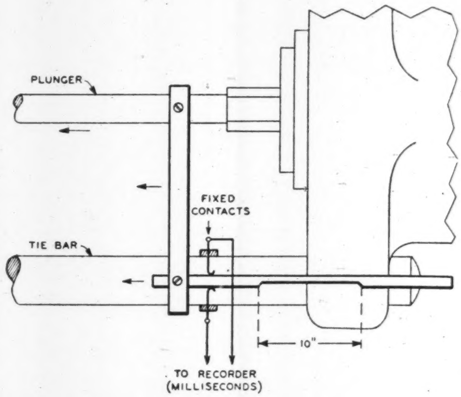


FIG. 8.—Schematic of Electronic Plunger-Speed Recording Unit.

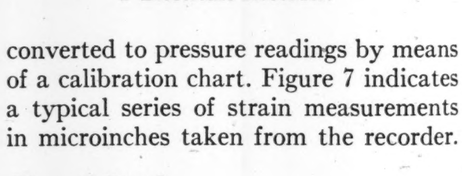


FIG. 9.—Typical Chart of Plunger Speed From Electronic Recorder.

Plunger Speed:

The measurement of plunger speed was accomplished with two sets of instruments. Figure 8 indicates a schematic diagram of an electronic type of speed-recording unit. This instrument consists essentially of a slide bar rigidly attached

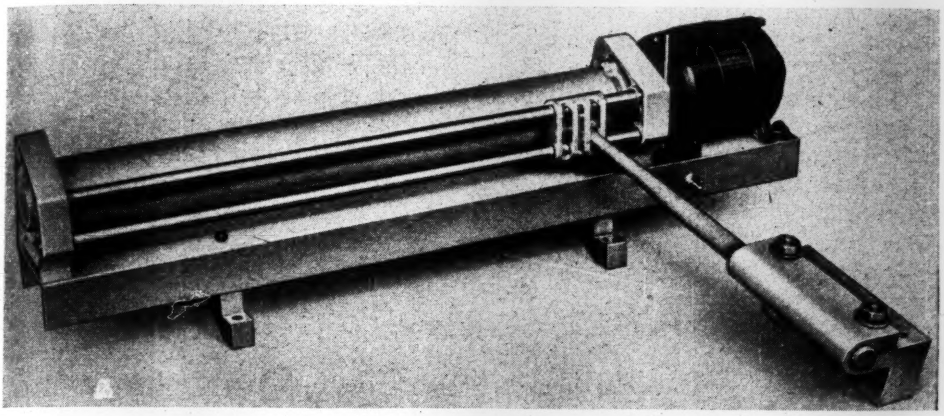


FIG. 10.—Drum-Type Plunger Speed Recorder.

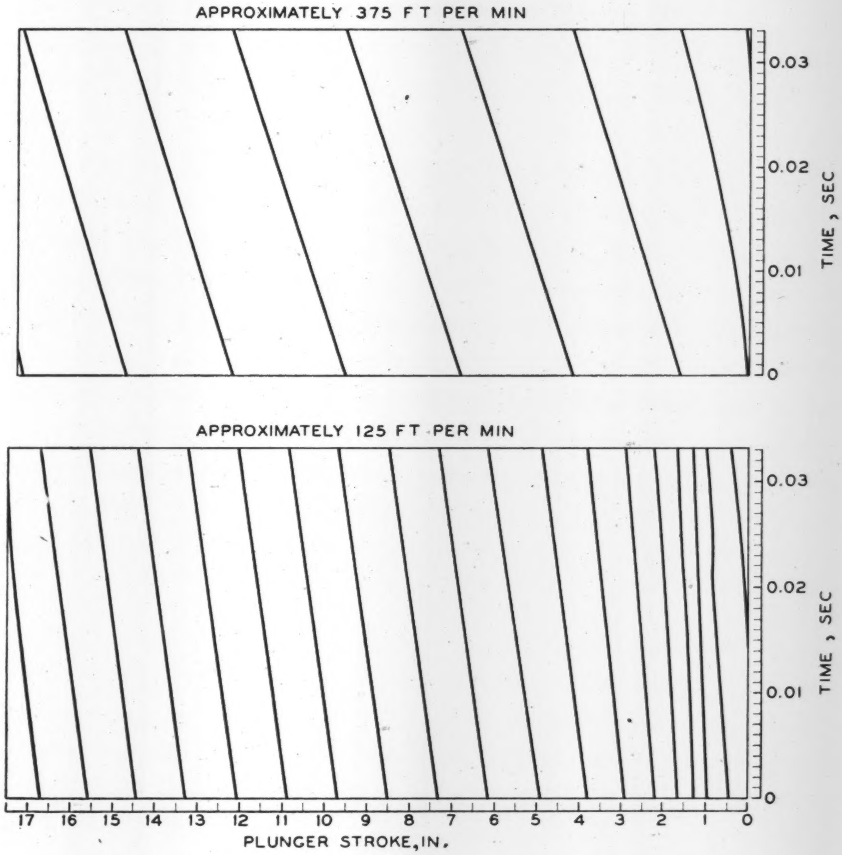


FIG. 11.—Typical Chart of Plunger Speed From Drum Recorder.

to the injection plunger, and passing through a switchbox containing contact members which in turn operate in a horizontal groove in the slide bar. This groove has an offset section approximately 10 in. long. When the plunger moves forward, it carries the slide bar with it and the contact operated by the offset groove actuates a Speedomax recorder during the time required for approximately 10 in. of plunger travel. While this instrument does not determine the total time required for a complete plunger stroke, it effectively measured the relative plunger speed during the period in which the speed is maximum and serves as an extremely useful tool for observing the uniformity of operation of the plunger. Figure 9 shows typical speed curves recorded on this unit.

Plunger speeds were also measured on a drum-type recorder (Fig. 10), capable of measuring the entire plunger stroke. A drum approximately 18 in. long is rotated directly from an 1800-rpm synchronous motor. The circumference of the drum is such that, when operating at 1800 rpm, each $\frac{1}{4}$ in. of circumferential travel is equivalent to 0.001 sec elapsed time. A sliding member carrying a stylus is connected by means of a rigid arm to the plunger. Prior to making a shot, a chart is placed upon the outside circumference of the drum. The drum is set in motion, and by means of an actuating lever the stylus is brought in contact with the chart paper. When the plunger moves forward carrying with it the slide member and stylus, a spiral curve is produced on the recorder paper. Upon removal of the paper from the drum, a complete analysis may be made of the plunger stroke, including total elapsed time, maximum plunger speed, and the rates of acceleration and deceleration of the plunger. Figure 11 shows typical curves taken from this type of recorder for high and low plunger speed. Since a separate chart is required on this recorder for each stroke of the plunger, it was not practical to obtain measurements on all castings. These determinations were made with this equipment on approximately every tenth casting.

Technique:

The following is a discussion of the techniques employed in making each of the test casting runs. In order to permit the production of castings at relatively low pressures, a special valving arrangement was installed in the hydraulic system of the die-casting machine. As a result, high pump efficiency and relatively uniform plunger speed was achieved at all pressures.

The original investigation, as planned, did not include variations in plunger speed or in the die venting. Early in the casting operations, however, it became apparent that commercial castings could not be produced under certain pressure conditions. It was deemed advisable, therefore, to widen the scope of the investigation to include both a high and a low plunger speed and to create an additional condition of venting in the die by the use of die shims, in order that under each pressure condition, castings could be produced of a sufficiently high quality to lend themselves to suitable mechanical testing.

At the start of each run, the required casting conditions were set up and castings produced until uniformity of operations had been achieved. All recording devices were then synchronized and the test castings representing the test lot were made and identified. By careful marking of all recording charts, each casting was correlated with the temperature, pressure, and speed condition under which it was made. A total of 43 test casting runs were made, employing two gate thicknesses, eight calculated

TABLE I.—SUMMARY OF CASTING CONDITIONS AND AVERAGE TEST VALUES.

Run	Gate Thickness, in.	Calculated Pressure, psi	Measured Pressure Total Run, psi			Measured Pressure 5 Test Castings, psi			Average Plunger Speed, ft per in	Die Shims, in.	Average Die Face Temperature, deg. Fahr	Average Quality Index (Complete Run)	Average Soundness Rating (5 Test Castings)	Maximum Pad Thickness Test Bar Section, in.	Tensile Strength, psi			Yield Strength, 0.2 per cent Offset, psi			Elongation in 2 in., per cent
			Max	Min	Avg	Max	Min	Avg							Max	Min	Avg	Max	Min	Avg	
1A(1-25)	0.090	1 850	2 550	1 650	2 050	2 150	1 900	2 100	129	-	620	0.6	3.8	0.290	25 500	16 700	21 500	15 800	14 100	15 000	2.1
1A(1-15)	0.090	1 850	2 550	2 000	2 200	2 150	2 050	2 100	131	0.010	520	4.3	6.6	0.292	33 100	27 300	31 000	17 800	17 500	17 700	2.5
1B(1-35)	0.090	3 000	2 300	1 400	1 800	2 300	1 650	1 950	130	-	540	1.3	6.0	0.290	30 000	23 900	27 200	17 400	16 200	16 800	3.2
1BX(1-15)	0.090	3 000	3 600	2 300	3 100	3 300	2 300	2 900	129	0.010	535	3.9	5.2	0.293	31 200	24 800	27 700	17 600	17 100	17 300	2.8
1BB(1-25)	0.090	3 000	3 400	2 800	3 050	3 300	3 000	3 100	372	-	535	1.7	3.4	0.290	29 300	23 000	26 000	15 900	15 200	15 600	2.9
1G(1-10)	0.090	3 700	3 700	2 650	3 300	3 700	2 650	3 350	127	-	500	3.0	6.0	0.293	33 500	26 000	29 700	18 300	17 400	17 800	3.1
1G(11-20)	0.090	3 700	3 850	2 200	2 850	3 850	2 450	3 100	127	0.010	505	2.7	4.6	0.293	31 900	22 700	27 400	18 000	17 500	17 700	2.6
1G(21-35)	0.090	3 700	3 600	3 200	3 400	3 500	3 200	3 400	367	0.010	520	5.3	7.4	0.295	34 500	32 000	33 400	18 400	17 900	18 100	3.1
1C(1-25)	0.090	6 000	4 200	3 100	3 650	3 800	3 200	3 550	128	-	530	2.2	6.8	0.292	30 000	23 000	26 600	17 500	16 600	17 000	2.2
1CC(1-25)	0.090	6 000	4 400	3 300	4 100	4 300	4 000	4 250	391	-	505	5.2	8.2	0.294	35 000	31 900	33 500	17 400	17 000	17 200	3.1
1CX(1-15)	0.090	6 000	4 950	3 500	4 200	4 950	3 500	4 200	129	0.010	490	3.2	5.4	0.294	32 800	25 300	29 000	18 300	17 700	18 000	2.7
1CX(16-30)	0.090	6 000	4 700	3 400	4 250	4 600	3 800	4 200	374	0.010	495	5.0	7.6	0.295	34 600	29 500	32 100	18 400	17 500	18 000	3.1
1H(1-15)	0.090	10 000	8 700	7 500	8 050	8 300	7 500	7 950	130	-	485	2.6	5.4	0.292	30 500	24 000	27 800	18 000	17 400	17 700	3.0
1H(16-30)	0.090	10 000	8 450	7 050	7 800	8 450	7 300	7 950	125	0.010	475	4.3	7.0	0.295	34 700	26 300	30 000	18 500	17 000	17 800	2.3
1H(31-50)	0.090	10 000	8 800	6 650	7 300	8 800	7 000	7 700	379	0.010	460	4.0	7.6	0.296	35 900	30 800	33 900	18 300	17 800	18 100	3.4
1D(1-25)	0.090	12 000	12 200	9 500	10 500	11 400	10 000	10 600	145	-	510	4.8	8.2	0.295	32 700	26 500	29 600	18 200	17 600	17 900	2.2
1DD(1-15)	0.090	12 000	11 000	9 000	9 900	10 350	9 000	9 800	130	0.010	490	8.0	9.8	0.301	35 400	29 700	32 400	18 700	18 000	18 300	3.1
1E(1-25)	0.090	24 000	20 200	18 200	19 000	20 200	18 550	19 100	136	-	500	9.5	10.0	0.304	33 400	29 100	31 600	18 700	17 500	18 100	2.8
1EE(1-15)	0.090	24 000	19 600	16 350	18 400	19 050	18 100	18 450	450	0.010	460	9.9	10.0	0.304	38 500	34 200	36 500	18 800	16 500	18 600	3.4
1EX(1-15)	0.090	24 000	21 200	19 500	20 500	21 000	20 000	20 550	126	0.010	475	8.9	9.8	0.303	32 200	23 200	28 000	18 800	17 900	18 400	3.0
1F(1-20)	0.090	48 000	40 500	37 100	38 250	40 500	38 000	39 050	124	0.010	465	7.4	10.0	0.316	28 700	22 500	25 400	18 500	17 700	18 100	1.8
1FF(1-6)	0.090	48 000	38 650	32 350	36 500	38 650	32 350	36 250	359	0.010	480	9.7	9.8	0.329	35 400	26 500	31 700	18 800	18 300	18 600	3.5
2A(1-25)	0.030	1 850	3 600	2 050	2 700	3 250	2 300	2 800	114	-	500	0.8	1.4	0.290	28 900	23 400	26 300	16 200	15 500	15 900	2.2
2A(26-37)	0.030	1 850	2 700	2 000	2 400	2 500	2 000	2 350	115	0.010	490	2.9	3.8	0.292	30 900	25 300	28 300	17 100	16 800	16 900	2.6
2B(1-25)	0.030	3 000	3 100	2 000	2 650	2 800	2 350	2 600	115	-	510	1.7	3.8	0.291	29 900	23 700	26 600	16 600	16 000	16 300	2.6
2B(26-40)	0.030	3 000	3 300	2 250	2 800	3 150	2 500	2 850	120	0.010	510	3.9	5.0	0.293	32 400	28 800	30 500	17 300	17 000	17 100	3.0
2G(1-12)	0.030	3 700	3 500	2 850	3 200	3 500	2 850	3 150	132	-	520	0.9	1.4	0.292	28 200	22 600	25 500	17 200	16 300	16 700	2.5
2G(13-27)	0.030	3 700	3 950	3 450	3 750	3 900	3 800	3 650	129	0.010	510	5.3	7.4	0.294	33 900	28 000	31 300	18 800	18 400	18 600	3.0
2G(28-42)	0.030	3 700	3 800	3 300	3 600	3 800	3 400	3 650	364	0.010	500	5.3	7.0	0.295	34 600	29 700	32 800	17 700	17 400	17 500	3.0
2C(1-15)	0.030	6 000	5 800	4 600	5 200	5 600	4 600	5 100	120	-	510	2.2	3.0	0.292	29 800	25 700	27 600	17 400	16 000	16 700	2.8
2C(16-30)	0.030	6 000	5 700	4 900	5 350	5 700	5 300	5 500	111	0.010	495	6.1	6.0	0.295	33 500	29 800	31 600	17 800	17 000	17 400	3.0
2C(31-40)	0.030	6 000	5 800	5 100	5 550	5 800	5 150	5 600	330	0.010	490	8.5	9.0	0.296	36 800	33 100	35 000	18 600	17 900	18 300	3.4
2H(1-15)	0.030	10 000	8 350	7 000	7 650	7 700	7 400	7 550	124	-	485	1.9	3.0	0.294	30 000	26 500	27 600	17 800	17 300	17 600	2.5
2H(16-30)	0.030	10 000	8 000	6 900	7 450	8 000	6 900	7 400	127	0.010	485	3.2	4.6	0.295	31 200	27 800	29 700	17 800	17 400	17 600	2.5
2H(31-45)	0.030	10 000	8 050	7 050	7 600	8 000	7 050	7 600	362	0.010	485	5.5	7.2	0.296	34 300	31 100	32 700	18 000	17 600	17 800	3.2
2D(1-25)	0.030	12 000	13 650	12 700	13 300	13 600	13 250	13 450	137	0.010	495	9.3	10.0	0.299	35 300	31 000	33 100	18 400	18 100	18 200	2.9
2DD(1-15)	0.030	12 000	13 550	12 100	12 850	13 400	12 650	12 950	140	-	510	5.1	7.8	0.299	34 700	29 300	32 100	19 300	18 400	18 900	3.1
2E(1-25)	0.030	24 000	25 000	23 400	24 300	25 000	23 850	24 300	136	0.010	505	9.9	10.0	0.305	36 200	28 100	32 300	19 100	18 500	18 800	3.3
2E(26-35)	0.030	24 000	24 300	23 400	23 850	24 300	23 400	23 750	365	0.010	470	9.8	10.0	0.304	37 300	33 500	35 500	19 000	18 500	18 800	3.7
2F(1-25)	0.030	48 000	52 300	47 300	49 400	52 000	48 100	50 000	125	0.010	465	8.8	9.6	0.320	29 400	23 900	27 200	17 800	17 500	17 700	2.4
2F(26-35)	0.030	48 000	52 700	48 000	49 550	48 900	48 000	48 450	405	0.010	460	9.7	10.0	0.317	36 600	33 200	35 200	19 200	18 800	19 000	2.6

pressures, two plunger speeds, and two conditions of die venting and are summarized as follows:

Gate Thicknesses, in.:

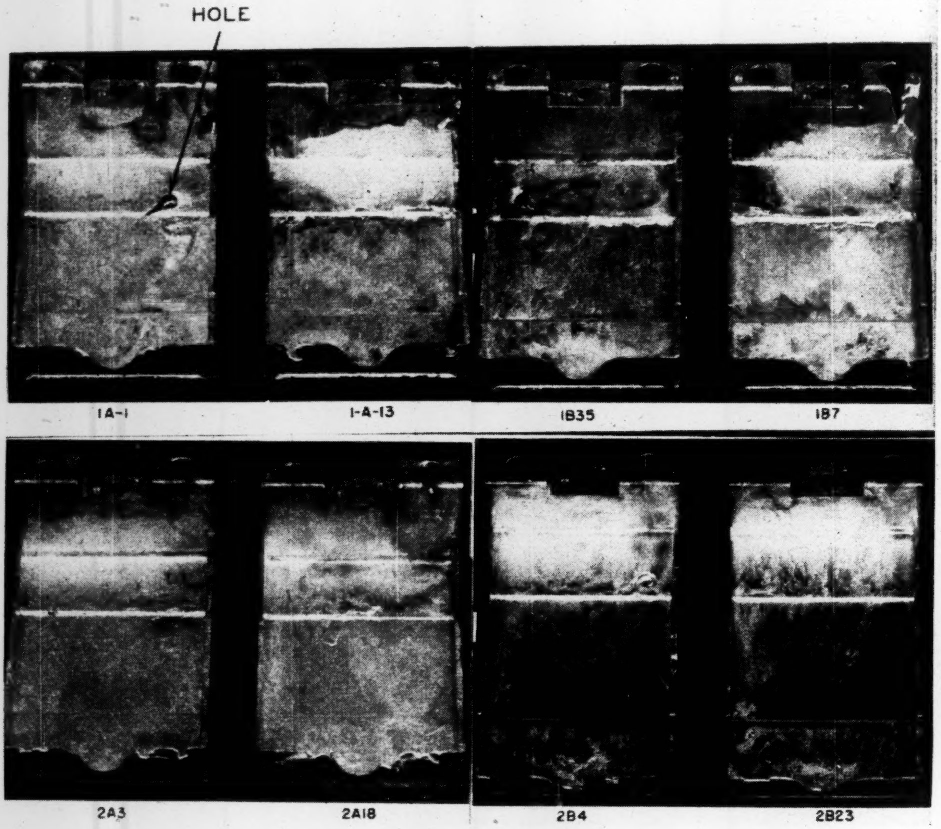
0.030
0.090

Venting:

No shims (normal 0.010 in. vent)
With shims (additional 0.010 in.)

By referring to Table I, the operating conditions for each run can be ascertained.

Low Pressure—0.090 in. Gate



Low Pressure—0.030 in. Gate

FIG. 12.—Typical Rejected Castings.

Calculated Pressures, psi:

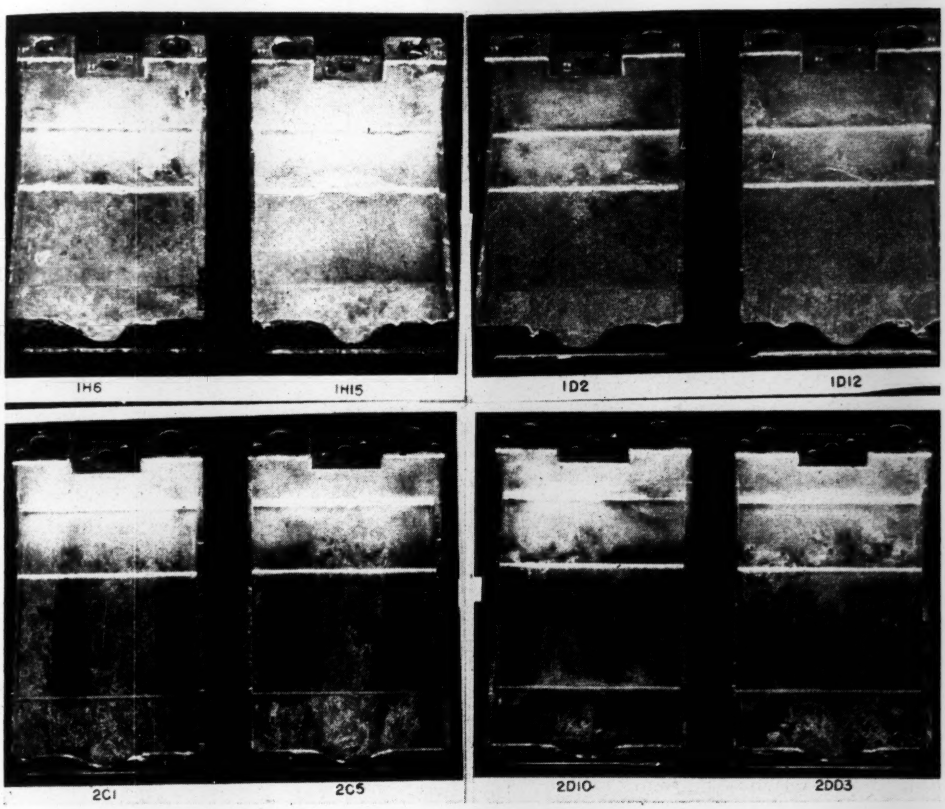
1 850
3 000
3 700
6 000
10 000
12 000
24 000
48 000

Plunger Speeds, ft per min:

Slow (approximately 125)
Fast (approximately 375)

Upon completion of the casting operations, the gross weight of each run of castings was determined, as a check on the soundness and completeness of filling. It was determined, however, that variations in the pad thickness in the test section of the castings due to shimming of the die and to springing of the dies under high pressure, produced weight variations which precluded the use of these values. The weight determinations, therefore, do not appear in this report since it is felt that they are of no great significance. The variations in pad thickness, however, are included in Table I as a matter of information.

Intermediate Pressure—0.090 in. Gate

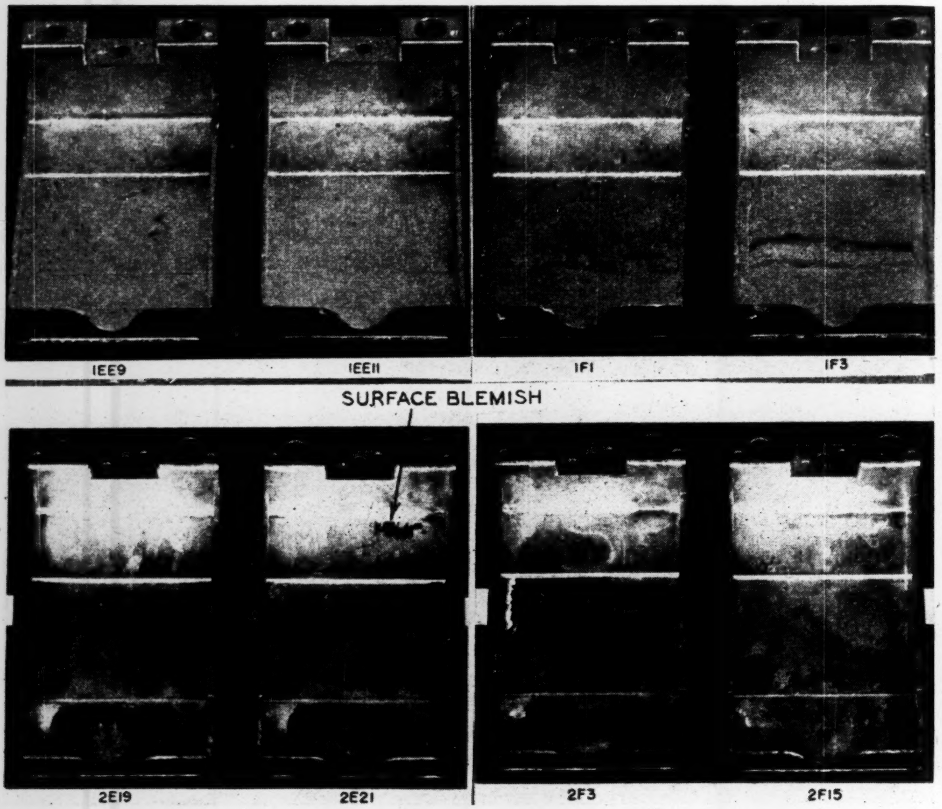


Intermediate Pressure—0.030 in. Gate
FIG. 13.—Typical Rejected Castings.

Each casting was subjected to a careful visual examination, in order to determine whether the castings were commercially acceptable from the standpoint of surface appearance and completeness of fill. Castings which did not come up to commercial standards were set aside for future reference. Photographs were taken of typical rejected castings and acceptable test castings. Figures 12, 13, and 14 show typical rejected castings at low, intermediate and high pressures, respectively. Similarly Figs. 15, 16, and 17 show typical acceptable test castings. Table II shows the results obtained from the visual examination of the castings, indicating the rejection rate for each casting condition.

Radiographs were then made of all acceptable test castings. A preliminary examination of the radiographs was made, in order to select those representing a range of soundness from the worst to the best condition. By this method, ten typical radiographs were selected as standards and were assigned numbers ranging from 1 to 10 in the order of increasing soundness. A composite radiographic standard was thus set up. The set of radiographic standards so produced is shown in Fig. 18. Using these standards as a guide, each radiograph of the test castings was examined and assigned a number from 1 to 10 corresponding to its relative soundness. Castings which had previously been rejected on the basis of visual examination were given a rating of zero. For each casting run, the porosity and rejection ratings were averaged to obtain a quality index, shown in Table I. The five castings possessing the highest soundness rating in each group were used for mechanical tests. The average soundness rating of these five selected castings is also shown in Table I.

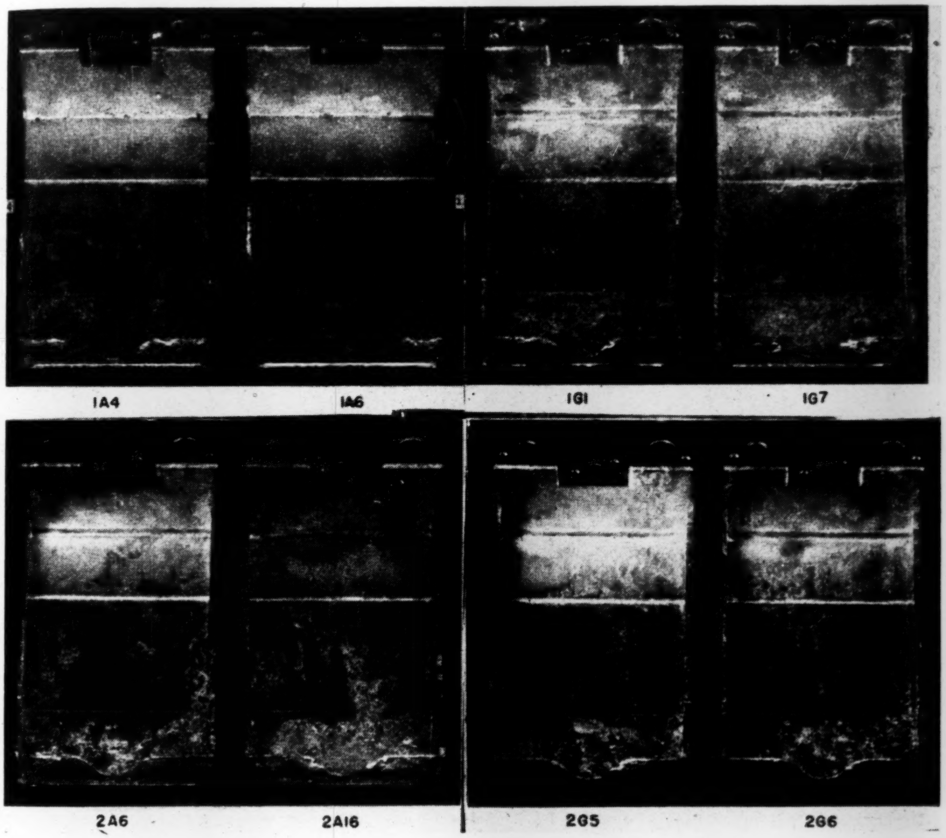
High Pressure—0.090 in. Gate



High Pressure—0.030 in. Gate

FIG. 14.—Typical Rejected Castings.

The test section of each casting thus selected was cut into four bars, the sides of which were milled parallel to produce flat-type tension test bars. In this manner, 20 test bars were prepared from each casting run. Each of the test bars was subjected to tensile strength and elongation tests, and 50 per cent of the bars to yield strength determinations. The maximum, minimum, and average values from these determinations are shown in Table I. It should be pointed out that considerable difficulty was encountered in these mechanical tests, due to the presence of "cold-shuts" in the fillet region of the upper pad. As a result, the elongation values were not obtainable in many cases. The elongation values shown in Table I are, therefore, the averages of all determinable results.


Low Pressure—0.090 in. Gate

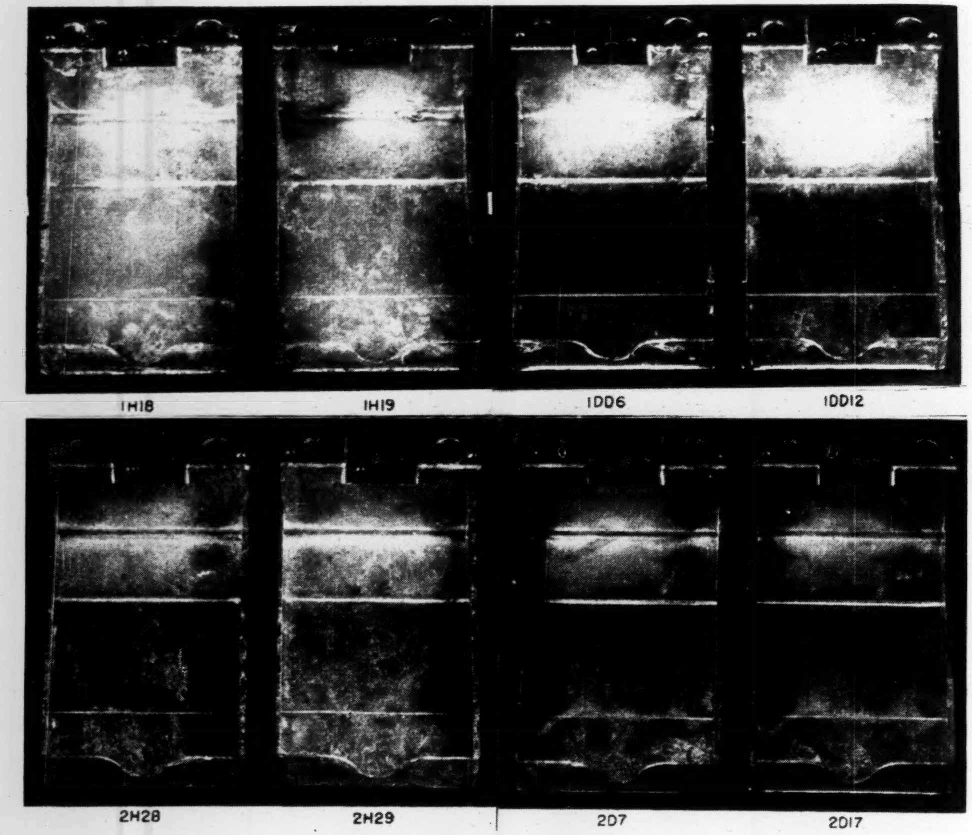
Low Pressure—0.030 in. Gate

FIG. 15.—Typical Acceptable Test Castings.

DISCUSSION OF DATA

The information contained in Table I is largely self-explanatory. It should be noted that the plunger-speed values indicated are the averages obtained from the drum-type recorder for each run. The column headed "Average Die-face Temperature" contains the values obtained by the Rayotube and electronic recorder. The temperatures determined by the four thermocouples located in the die block and the thermocouple in the pouring port are too voluminous for inclusion in this report. However, they indicated that the desired control conditions in the die were obtained throughout each run and that extremely uni-

Intermediate Pressure—0.090 in. Gate



Intermediate Pressure—0.030 in. Gate

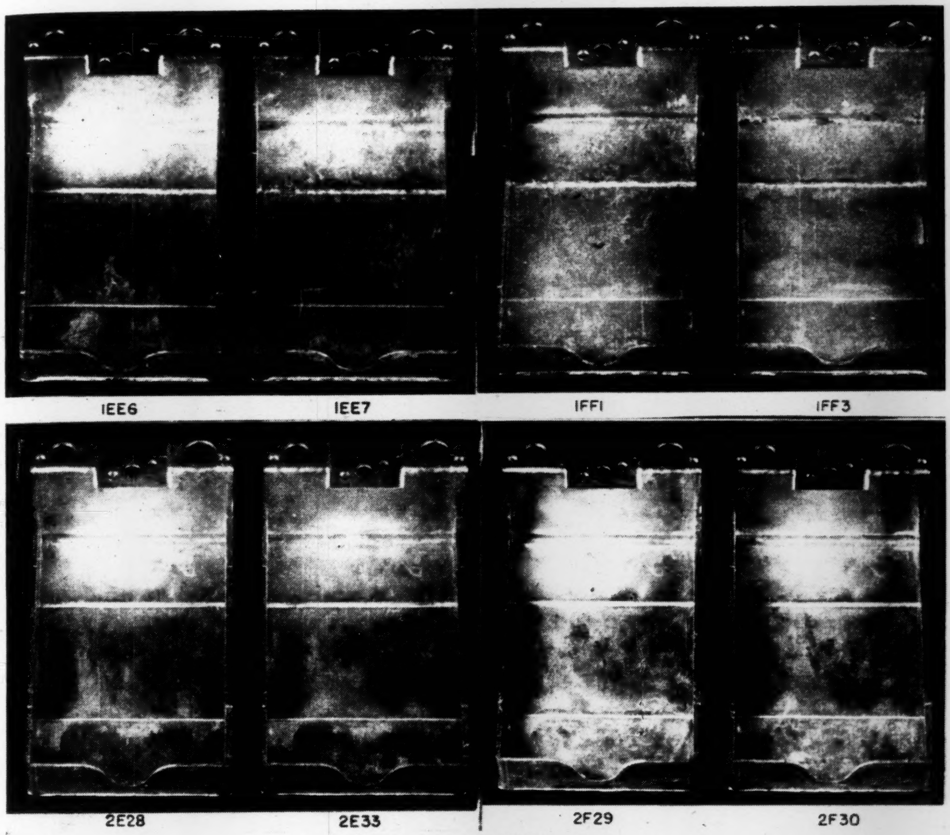
FIG. 16.—Typical Acceptable Test Castings.

TABLE II.—RESULTS OF VISUAL INSPECTION.

Calculated Pressure, psi	Rejection Rate, per cent of Total Run							
	Series No. 1—0.090 in. Gate				Series No. 2—0.030 in. Gate			
	Condition 1	Condition 2	Condition 3	Condition 4	Condition 1	Condition 2	Condition 3	Condition 4
	Slow Plunger No Shims	Slow Plunger With Shims	Fast Plunger No Shims	Fast Plunger With Shims	Slow Plunger No Shims	Slow Plunger With Shims	Fast Plunger No Shims	Fast Plunger With Shims
1 850.........	76	0	[a]	[a]	32	0	[a]	[a]
3 000.........	68	0	8	[a]	12	0	[a]	[a]
3 700.........	50	30	[a]	13	25	7	[a]	0
6 000.........	52	27	12	0	7	0	[a]	0
10 000.........	40	20	[a]	30	13	0	[a]	0
12 000	28	13	[a]	[a]	0	0	[a]	[a]
24 000.........	8	0	[a]	0	[a]	0	[a]	0
48 000.........	[a]	20	[a]	0	[a]	0	[a]	0

[a] No castings were produced under these conditions.

High Pressure—0.090 in. Gate


1EE6
1EE7
1FF1
1FF3
2E28
2E33
2F29
2F30

High Pressure—0.030 in. Gate

Fig. 17.—Typical Acceptable Test Castings.

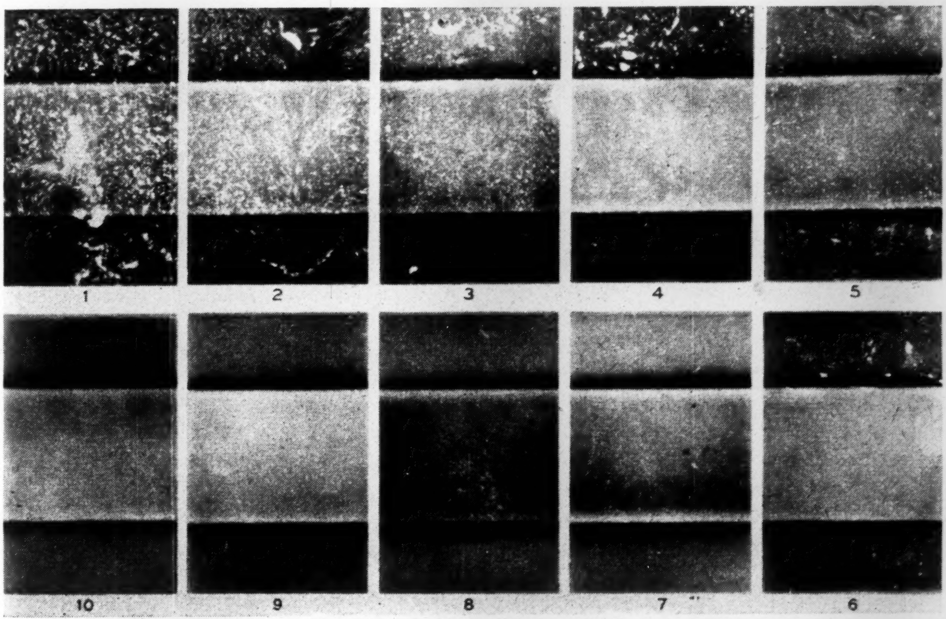

1
2
3
4
5
10
9
8
7
6

Fig. 18.—Radiographic Standards.

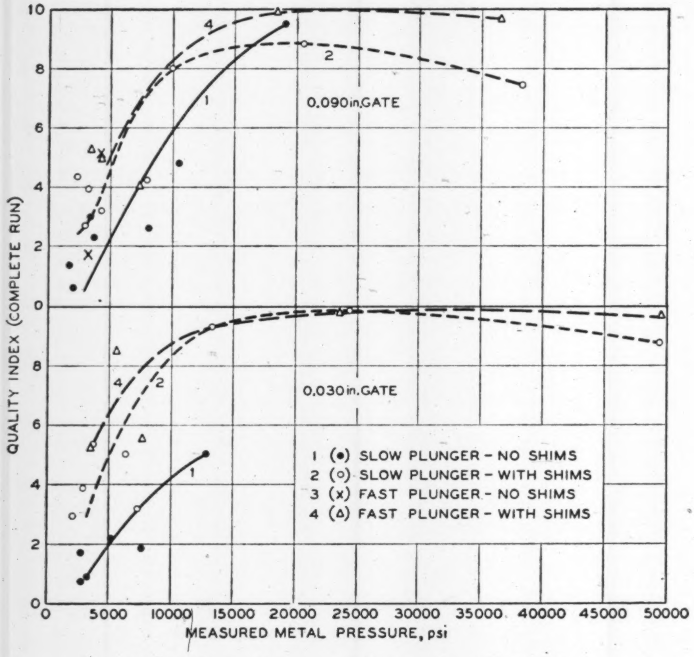


Fig. 19.—Effect of Measured Pressure on Quality Index.

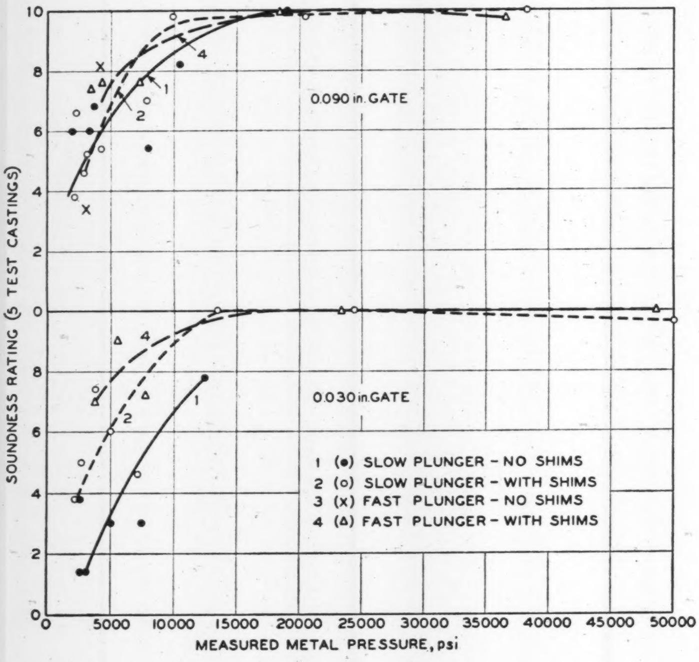


Fig. 20.—Effect of Measured Pressure on Soundness Rating.

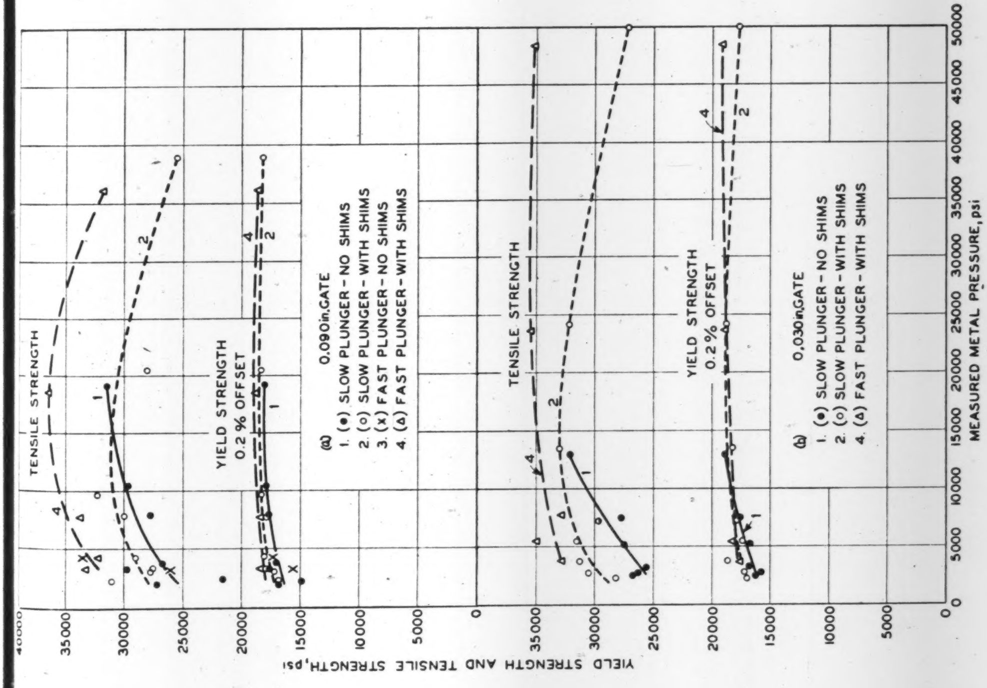


FIG. 21.—Effect of Measured Pressure on Mechanical Properties.

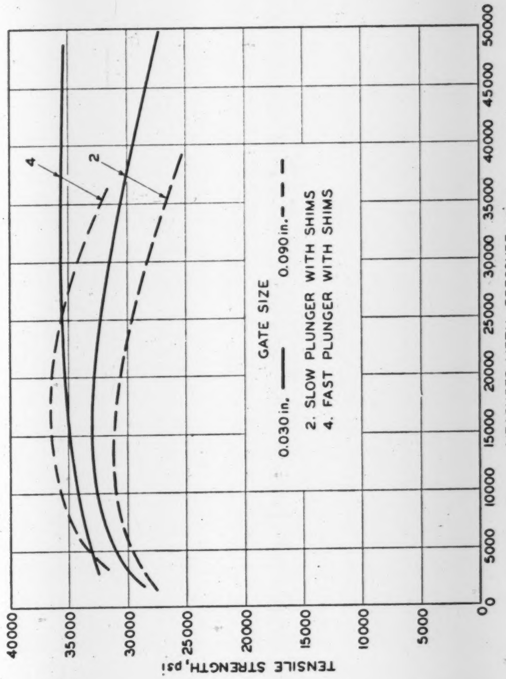


FIG. 22.—Effect of Measured Pressure on Tensile Strength.

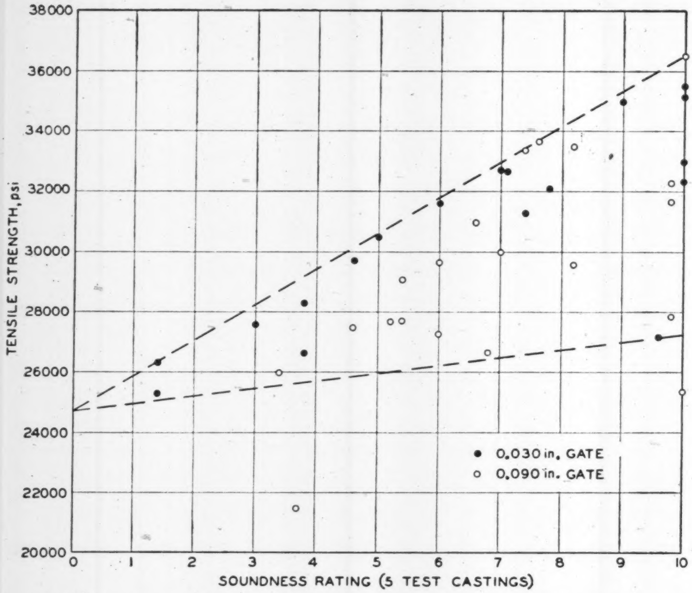


FIG. 23.—Variation of Soundness Rating With Tensile Strength.

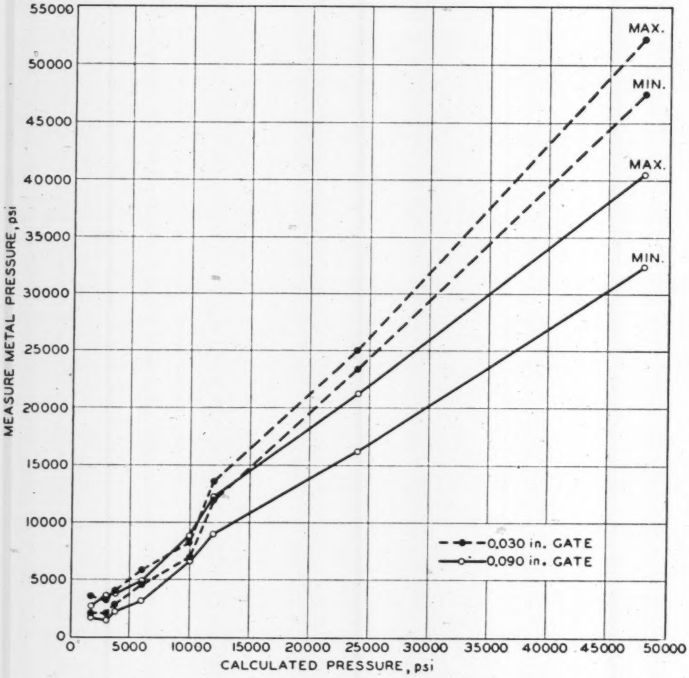


FIG. 24.—Comparison of Calculated Pressure With Measured Pressure.

form die temperatures prevailed for each casting condition. It should be appreciated that Table I represents a condensation of all the individual data obtained from the temperature, pressure, and plunger-speed recordings made throughout the entire castings series. Such a summary is both advisable and necessary for a concise presentation of the results and to permit a clear-cut analysis of the findings of the investigations.

The results of the visual inspection shown in Table II are based on calculated pressures, as a matter of convenience. It is recognized that the actual pressures obtained for a given calculated pressure are not identical for both gate sizes. An examination of these data shows that in general the rejection rate under all conditions was higher among castings produced with a 0.090-in. gate than those made with a 0.030-in. gate. It is also evident that castings produced with high plunger speeds were superior, visually, to those made with low plunger speeds. Generally, the additional venting provided by die shims reduced the rejection rates. The optimum visual quality, therefore, is achieved by employing a thin gate, fast plunger, and adequate venting.

Figures 19 to 24, inclusive, present a comparison of the mechanical test values, quality indices, and radiographic soundness ratings with various conditions of gating, venting, plunger speed and metal pressure. Figure 19 indicates the effect of measured metal pressure on the quality index for castings produced with both the thick and thin gates, at two plunger speeds, and under two conditions of venting. It may be seen that the information is incomplete for some of the conditions indicated. This is due to the fact that it was not possible to produce castings suitable for tests under some of these conditions. It may be observed that in the low-pressure areas, there is considerable scatter in the data. However, a general trend is indicated, showing that the quality index improves rapidly with increased metal pressure, reaching a maximum at approximately 20,000 psi. Beyond this point, no substantial improvement in quality index is obtained. In fact, it can be observed that castings produced with a slow plunger and a die having 0.010 in. shims, with both gate thicknesses, show a substantial reduction in quality index towards the upper end of the pressure range. It is also indicated that castings produced with a slow plunger, under both gate thicknesses, have a lower index of quality than those produced with a fast plunger. A comparison of curve No. 2 in Figs. 19(*a*) and (*b*), shows that castings produced with a slow plunger using a 0.030-in. gate possess a higher quality index than those produced with a 0.090-in. gate at a similar plunger speed. On the basis of quality index, therefore, these curves show that the best conditions are obtained when using a fast plunger, a gate thickness of 0.030 in., and adequate die venting. If it is assumed that a quality index of five or better were necessary for commercial acceptance of castings, these curves indicate that under these casting conditions, a minimum measured metal pressure of 5000 psi is required.

Figure 20 indicates the effect of measured metal pressure on the soundness ratings obtained on the five castings selected for test purposes. In most respects these curves are comparable to those in Fig. 19, showing a relatively higher level of soundness, inasmuch as the five samples were chosen on the basis of preferred radiographic rating. It is worthy of note that the same indication of an optimum measured pressure, in the order of 20,000 psi, is indicated on these curves. An examination of the data obtained at the lower pressures shows a number of values having a soundness rating below six. An arbitrary soundness rating of six was set as the minimum for commercially acceptable castings. Under such a classification, commercially acceptable castings could not be produced using some of these casting conditions.

The effect of measured metal pressure on the mechanical properties of the castings is shown on Fig. 21. The tensile strength curves indicate approximately the same relationship as has been previously brought out in the comparison of metal pressure with quality index in that maximum strength is achieved at approximately 20,000-psi metal pressure. The data obtained on castings made with a 0.090-in. gate at both plunger speeds and also those made with a 0.030-in. gate at low plunger speed, with adequate venting, shows some reduction in tensile strength at the upper end of the pressure range. Under these conditions, the level of tensile strength at the high end of the pressure range is no better than that obtained at the lowest pressures. While the maximum tensile strength is observed from castings made with a 0.090-in. gate, fast plunger, and adequate venting, it is felt that castings made with a 0.030-in. gate under similar conditions are more desirable because their mechanical properties are more uniform throughout the pressure range.

An examination of the yield strength curves in Fig. 21 shows no significant difference in yield strength under any of the conditions. These curves bear approximately the same relationship to each other as the tensile strength curves under comparable conditions. The results confirm previous experience indicating that yield strength values are relatively insensitive to casting conditions and are more suitable as an index to basic metal quality.

Figure 22 is a composite of curves No. 2 and No. 4 from Fig. 21 for the purpose of making a more direct comparison of measured metal pressure with tensile strength for castings made with both gate thicknesses, both plunger speeds, and with adequate die venting.

Figure 23 shows the tensile strength values plotted against the soundness ratings, differentiating only between castings produced with the two gate thicknesses. The purpose of this analysis is to determine the degree to which the mechanical strength of castings may be predicted from the radiographic examination. These data show that while there is considerable scatter, there is, as anticipated, a definite trend indicating an increase in tensile strength with improved soundness rating. It is also observable that castings made with the 0.030-in. gate are generally higher in tensile strength for equivalent soundness ratings than those produced with the 0.090-in. gate. With the exception of two test values, not readily explainable, all points fall within a relatively narrow band. The upper and lower boundary lines are for the purpose of indicating the maximum and minimum tensile strengths which may be anticipated for a given soundness rating.

A comparison of calculated pressure and maximum and minimum measured metal pressure for the two gate thicknesses is shown in Fig. 24. These curves show that substantially higher measured pressures were obtained for given calculated pressures when the 0.030-in. gate thickness was used and that there was substantially less variation between maximum and minimum values at a given pressure when using this gate. It is interesting to observe a vertical displacement in each of the curves at 12,000 psi calculated pressure, at which point the prefill system of the die-casting machine was first employed to obtain the higher pressures. It is obvious from an examination of these data that, when using the 0.090-in. gate, the metal pressure is substantially less than the calculated value. On the other hand, when using the 0.030-in. gate, the metal pressure is approximately equal to the calculated pressure. It is probable that the 0.030-in. gate restrains the metal flow sufficiently to produce a pressure build-up between the plunger and the gate, whereas the thick gate provides less restriction, and with a well-vented die cavity, the total potential pressure is not realized. In examining these data, it should be borne in mind that the calculated pressure values were based on readings from standard hydraulic pressure gages, not intended as precision instruments, using a multiplying factor of either 3.7 or 16.7, depending upon whether the single cylinder or the dual cylinders of the prefill system were employed. This point is brought out in order that no great significance be attached to the fact that in some instances actual measured pressures were slightly higher than the calculated values. The inability to obtain precise gage measurements on the hydraulic system at the extremely low pressures probably accounts for the unusual scatter of points in that area. These curves are of academic interest only and relate to the development of the method for obtaining measured metal pressures within the die.

A discussion of the conclusions which may be drawn from this investigation needs to be prefaced by some comment upon the various factors which are peculiar to this particular study. The die used in this study was intentionally designed to incorporate many of the problems common to die casting. The flat bottom section of the castings from which the test bars were cut contains two fillets which in effect are secondary gates. Intentional short shots made for the purpose of studying the flow of metal in this die indicated that the flow depends upon the thickness of gate employed. When using a 0.090-in. gate, the flow of metal appeared to take two directions. The majority of the metal flowed up the two side-wall sections of the castings to the thick cored sections at the upper end. Following the filling of the thick section, the metal pursued a course downward toward the gate through the pad section. Simultaneously, but at a lower rate, metal flowed directly up the pad section from the gate and the two paths of metal met in the pad section, usually at the top fillet. This flow pattern consistently produced serious cold shuts at the upper pad fillet and impaired the mechanical properties of the test castings.

Castings produced with the 0.030-in. gate showed a somewhat different flow of metal. As in the case of the 0.090-in. gate, the metal flowed up the two side-wall sections of the castings into the cored end section and thence down through the pad area to the lower end of the castings. This condition resulted in a joining of the metal paths at the lower end of the pad section, sometimes at the fillet but more often at the thick section near the gate.

The feasibility of studying metal flow within a die-casting cavity has been previously explored by the subcommittee. A number of discussions of this problem with the manufacturers of X-ray equipment disclosed practically insurmountable obstacles to a radiographic approach. The possibility of employing transparent dies was also found to be impractical. There is, therefore, at this time no completely satisfactory technique for the study of metal flow under pressure within the die.

CONCLUSIONS

Based upon the results obtained from this study, and subject to the limitations of the conditions covered, the following conclusions are drawn:

1. Satisfactory instrumentation has been developed for the measurement of die-casting process variables and include:
    (*a*) Metal-pressure-measuring system employing SR-4 strain gages on pressure cylinders and recorded on direct reading electronic recorders.
    (*b*) Die-face temperature measurement by means of the Leeds & Northrup Rayotube and Speedomax Recorder.
    (*c*) Plunger-speed measurement, employing both a Leeds & Northrup recording unit and a Western Electric Co. drum-type recorder.
2. This instrumentation is eminently practical and can be used to control the die-casting process.
3. The results show that the test die produced castings having the highest quality under the following conditions:
    (*a*) Measured metal pressure—20,000 psi,
    (*b*) Thin gate (approximately 0.030 in.),
    (*c*) High plunger speed (approximately 375 ft per min.),
    (*d*) Adequate die venting.

    Some deterioration in quality occurs at extremely high pressures. A minimum measured pressure of approximately 5000 psi is required to produce castings considered "commercially acceptable."
4. Castings made with a 0.030-in. gate had consistently higher quality indices and mechanical properties, under comparable casting conditions than those made with a 0.090-in. gate. It should be borne in mind that the comparison has been made between extremely thin and thick gates. On this basis, frequent reference has been made as to the superiority of the 0.030 in. gate over the 0.090 in. gate. Since no other gate sizes were studied, it should not be construed from these data that 0.030 in. is the optimum gate thickness.
5. The soundness and strength of castings made with a high plunger speed are superior under comparable conditions of pressure and venting to that of castings made with a slow plunger.
6. The thickness of die gate appears to be a factor in the relationship between calculated pressure and measured metal pressure.
7. A relationship is established between radiographic soundness and tensile strength by which maximum tensile strength may be predicted from soundness ratings.
8. Further work along the lines of this investigation should include:
    (*a*) Refinement of the test casting die to improve gating and venting.
    (*b*) Measurement of metal pressure at points within the actual die cavity.

Acknowledgment:

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ern Electric Co. for permission to employ the drum-type plunger speed recorder; J. R. Boettler of the Bell Telephone Laboratories for assistance in developing the metal-pressure measuring system; A. L. Jamieson of the Frankford Arsenal and J. R. Townsend of the Bell Telephone Laboratories for their advice in conducting the study and their helpful criticisms of this paper; The Ruge-DeForest Laboratories of Boston and the Baldwin-Southwark Co. of Philadelphia for permission to employ the SR-4 strain gage in this work.

DISCUSSION

MR. G. L. CHERRY.[1]—I think there is one thing that might be given a little more emphasis. The figures given in this paper are applicable to a particular casting. We recently had experience with an aluminum die casting where we found it necessary to use a plunger speed of 585 ft per min to obtain good parts. A companion casting to this was practically all junk when run at this speed, but good parts were produced at about 250 ft per min. On the other hand no good castings were produced of the first part when run at the slower plunger speed.

I believe we are unable to generalize to the extent of stating that a certain gate size, plunger speed, and casting pressure are applicable to all castings.

MR. J. C. FOX.[2]—The authors of this paper are to be congratulated for the time and effort put into it. This is probably the first report published on the control of the variables of the die-casting process, and undoubtedly this paper will stimulate the presentation of more papers of its kind on this subject.

There is developed in this paper some basic information which has not been previously available and which should be of great value in the development of better and higher quality die castings.

While there may be some controversy over the conclusions drawn by the authors, the results of their tests indicate clearly that both the strength and soundness of a die casting are improved by increased metal pressure which is quite well established. There is no doubt that any such controversial questions will be easily resolved, especially when others publish the results of their work on this problem.

MR. BRUNO SACHS[3] (*by letter*).—The committee—and, in particular, Messrs. W. Babington and D. H. Kleppinger—are to be congratulated for this excellent, data-packed report and for the condensed and clear presentation of the results of such a large amount of complex and systematic experimental work.

The authors explain that a special valving arrangement was installed in the hydraulic system of the die-casting machine. As a result, high pump efficiency and relatively uniform plunger speed was achieved at all pressures. Figure 11 shows two typical charts of the plunger speed from the drum recorder. It can be seen that the plunger stroke is 17½ in. The actual cavity filling of the die under consideration takes place during the last 3.15 in. of the stroke (assuming a cold chamber diameter of 2 in. as indicated in Mr. Jacobi's paper to which the authors refer).

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[1] Engineer, Foundry, Die Casting and Woodworking, Western Electric Co., Inc. Chicago, Ill.

[2] Chief Metallurgist, Doehler-Jarvis Corp., Toledo, Ohio.

[3] Quiet-Heet Mfg. Corp., Newark, N. J.

The question which I should like to ask is whether this constant speed was observed uniformly on all the various runs. It should be borne in mind that in the plunger speed chart for 125 ft per min, for instance, a change of the angle of only 3 deg represents a velocity change of approximately 40 per cent.

It would be interesting to know the length of the residue and whether all runs were made with the 2-in. diameter cold chamber; also, how the quality index was calculated.

Assuming constant plunger velocity for the die filling period, it can be shown—based on fundamental laws of mechanics, hydro- and thermo-dynamics,—that the following relationships do exist:

where:

f_{v0}, f_0, w_0, f_k, x_3, x_2, F, L_0, f, $q = q'$, f_0', c', c . . . see attached Figs. 25 and 26.

T_1 = absolute temperature of air in cavity and cold chamber for plunger position $x = x_1$, (see Fig. 26),

P_0 = pressure in accumulator at the beginning of the shot,

μ_0 = plunger friction coefficient,

p_{at} = atmospheric pressure,

γ = density of molten metal,

g = gravity acceleration,

ξ_0 = sum of resistance coefficients of all resistances in metal system, referred to plunger velocity, w_0,

ρ = density of drive fluid,

ξ' = sum of resistance coefficients of all resistances in return system, referred to velocity c', and

Optium vent area, $f_{v0} = \dfrac{f_0 w_0}{20.6\sqrt{T_1}}$

Plunger velocity, $w_0 = \sqrt{\dfrac{0.97 f_k P_0 \left[1 - \dfrac{\alpha}{2}(x_3 + x_2) - \beta\right]}{C_0 + C_1}}$

Porosity, $m = \left[\dfrac{1}{0.53}\,\dfrac{f_0(1 + 1.15\mu_0)p_{at}}{0.97 f_k P_0(1 - \alpha x_3)}\right]^{\frac{1}{7/5(1-n)}}$

Accumulator factor, $\alpha = \dfrac{1.4 f_k}{F L_0}$

Venting factor, $n = \dfrac{18.6 f_{v0}\sqrt{T_1}}{f_0 w_0}$

Cavity air pressure factor, $\beta = \dfrac{1 - \alpha x_3}{\frac{7}{5}(1 - n) - 1}\,m - \dfrac{f_0(1 + 1.15\mu_0)p_{at}}{0.97 f_k P_0}\left[\dfrac{1}{0.53[\frac{7}{5}(1 - n) - 1]} + 1\right]$

Metal system characteristic (die and operating condition), $C_0 = \dfrac{\gamma}{2g} f_0(1 + 1.15\mu_0)\left[\left(\dfrac{f_0}{f}\right)^2 + \xi_0 - 1\right]$

Machine Characteristic, $C_1 = \dfrac{\rho}{2g}\,\dfrac{1}{q^2}\,[(1.03 f_k - 0.88 f_0')(f_k - f_o')^2(1 + \xi') + 0.97\, f_k^3 \xi]$

ξ = sum of resistance coefficients of all resistances in drive system, referred to velocity c.

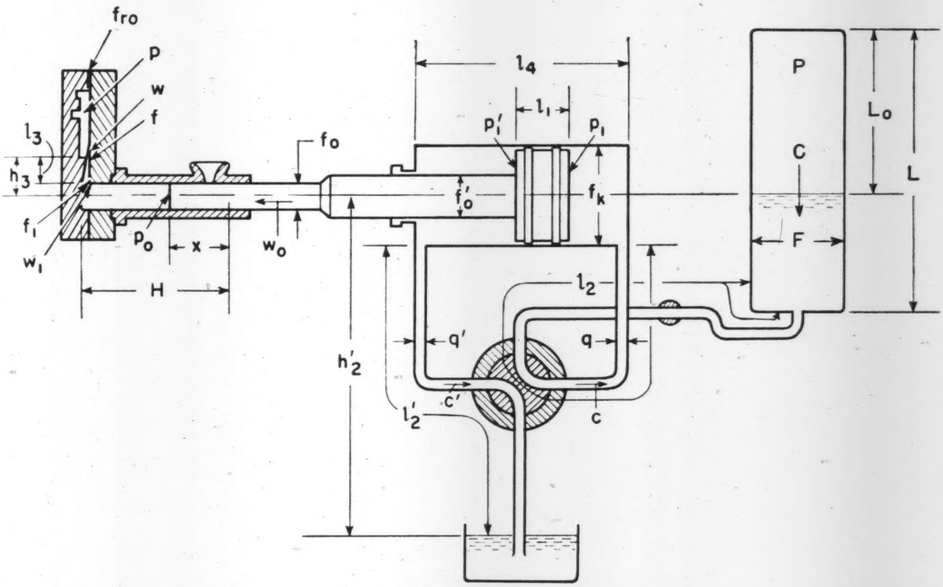

Fig. 25. Cold Chamber Machine Setup.

x = plunger displacement at time t, measured from plunger start $x = 0$, $t = 0$
F = area of accumulator
f_k = area of piston
f_0' = area of piston rod
f_0 = area of plunger
f_1 = area of gate entrance
f = area of gate exit
f_{v0} = area of vent slots in die
q = area of drive pipe line
q' = area of return pipe line
P = pressure in accumulator
p_1 = pressure on piston area f_k
p_1' = pressure on piston area $(f_k - f_0')$
p_0 = pressure on plunger area f_0
p = pressure of air in die cavity
L = length of accumulator
L_0 = height of nitrogen in accumulator at $t = 0$
l_2 = length of drive pipe line
l'_2 = length of return pipe line
l_1 = length of piston
l_3 = length of gate
l_4 = length of cylinder
h'_2 = height of plunger axis above reservoir level
h_3 = height of gate exit above plunger axis
H = distance of plunger at $x = 0$ to ejector half of die
C = velocity of drive fluid in accumulator
c = velocity of drive fluid in drive pipe line
c' = velocity of drive fluid in return pipe line
w_0 = velocity of piston
w_1 = velocity of metal in gate entrance
w = velocity of metal in gate exit

An analysis of these equations in conjunction with the condition of the experimental runs is in agreement with points 3 (*b*), 3 (*c*), and (6) of the conclusions in the report. A further analysis reveals that the quality of castings produced on hydraulically driven plunger machines (in contrast to air driven machines) is relatively insensitive as to gate thickness. Test runs (Table I) 1E (1–25), 1 EE (1–15), and 1 EX (1–15) with 0.090 in. gate thickness confirm this and it would seem that these particular test runs were not given sufficient consideration in the conclusion.

Conclusion 3 (*a*) as to the metal pressure refers to the test conditions. It should be possible to produce equally satisfactory castings with other pressures, provided temperature, gate, velocity, and venting conditions are properly coordinated.

There are no further comments except that the report represents a great step forward toward a better understanding of the die-casting process.

I should like to join the authors in their recommendation as to further work (conclusion 8) and to add that this should be done within the framework of a working theory in order to extend the results to various types of die-casting machines, dies, and casting conditions.

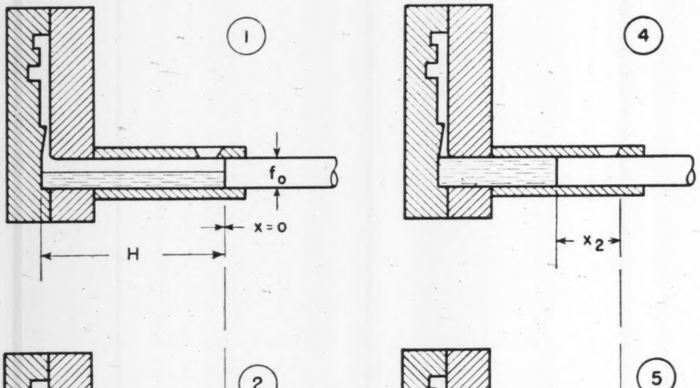

Fig. 26. Plunger Positions in Cold Chamber.

Mr. D. H. Kleppinger (*author's closure*).—I agree with Mr. Cherry's comment on our paper that each casting may be an individual problem, and because of this, overall generalizations cannot be made. It was hoped that this factor had been made clear in the conclusions of the paper where it was stated that the results obtained from the study were limited to the conditions covered.

This paper was not intended to be a "cure-all" for the die casting industry; and no one ever entertained the idea that data would be generated which would enable a die caster to consult a group of tables, charts, etc., set up his machine and produce good castings. It was intended to be a systematic scientific approach to determine the effects of some of the process variables on the properties and quality of castings.

The authors wish to thank Messrs. Fox and Sachs for their encouragement and constructive criticism. Mr. Sachs remarked that the filling of the die cavity took place in the last 3.15 in. of the stroke assuming a cold chamber diameter of 2 in. The diameter of the cold chamber was $1\frac{3}{4}$ in. and the filling of the cavity would take place in the final 3.8 in. of the stroke.

The special valving which was installed in the hydraulic injection system did result in high pump efficiency, resulting in plunger speeds which were within eight per cent of the nominal values quoted for all pressure runs.

In all of the casting runs, attempts were made to hold the length of the residue of "biscuit" to $\frac{3}{4}$ in. There were of course, some minor variations from this length, but it is felt that they are insignificant.

The quality index was calculated in the following manner: All of the castings were radiographed, the radiographs were scanned and a group of ten typical radiographs representing a range of soundness from the worst to the best conditions were selected as standards and were assigned numbers from one to ten in the order of increasing soundness. Each test casting within a casting run was then compared to the standards and given its rating, the rating of zero being given to castings which were originally rejected on visual examination. The rating numbers were added together for a run and divided by the number of castings within the run to give a figure which was termed "quality index."

Mr. Sachs' comparison of data obtained in the tests with theoretical values computed by formula which are developed in his forthcoming paper is interesting.

This paper is at the present time under study by a task group of Subcommittee IX of Committee B-6, and the authors desire to with-hold comment on these comparisons until his paper is published.

REPORT OF COMMITTEE B-7
ON
LIGHT METALS AND ALLOYS*

Committee B-7 on Light Metals and Alloys held two meetings during the year: one at Atlantic City, N. J., in June, 1950, and one at Cincinnati, Ohio, in March, 1951.

The committee consists of 75 members of whom 71 are voting members; 35 are classified as producers, 29 as consumers, and 11 as general interest members.

The committee wishes to express its sorrow and regret at the passing of J. J. Bowman who was secretary of the committee from June, 1944, until his death.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1950 Annual Meeting, Committee B-7 presented, to the Society through the Administrative Committee on Standards the following recommendations:

Revision of Tentative Specifications for:

Aluminum-Base Alloy Sand Castings (B 26 - 50 T),
Aluminum-Base Alloy Permanent Mold Castings (B 108 - 50 T),
Aluminum and Aluminum-Alloy Sheet and Plate for Use in Pressure Vessels (B 178 - 50 T), and
Aluminum-Base Alloys in Ingot Form for Sand Castings, Die Castings, and Permanent Mold Castings (B 179 - 50 T).

These recommendations were accepted by the Standards Committee on September 26, 1950, and the revised tentative specifications appear in the 1950 Supplement to Book of ASTM Standards, Part 2.

REVISION OF TENTATIVES

Committee B-7 recommends that Tentative Specifications for Aluminum and Aluminum-Alloy Sheet and Plate for Use in Pressure Vessels (B 178 - 50 T)[1] and Tentative Specifications for Aluminum and Aluminum-Alloy Sheet and Plate (B 209 - 50 T)[1] be revised by changing the lot size for tension tests from "1000 lb of sheet and 2000 lb of plate" to "2000 lb and 4000 lb," respectively. The committee also recommends that B 209 be revised by the deletion of Footnote *b* of Table II, renumbering subsequent footnotes accordingly.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends that the following tentative specification be continued without revision:

Tentative Specifications for:

Magnesium-Base Alloy Sand Castings (B 80 - 49 T),
Magnesium-Base Alloy Sheet (B 90 - 49 T),
Magnesium-Base Alloy Forgings (B 91 - 49 T),
Magnesium-Base Alloys in Ingot Form for Sand Castings, Die Castings, and Permanent Mold Castings (B 93 - 49 T),
Magnesium-Base Alloy Bars, Rods and Shapes (B 107 - 49 T),
Magnesium-Base Alloy Permanent Mold Castings (B 199 - 49 T),
Aluminum and Aluminum-Alloy Bars, Rods, and Wire (B 211 - 49 T),
Magnesium-Base Alloy Extruded Round Tubes (B 217 - 49 T),
Aluminum and Aluminum-Alloy Extruded Bars, Rods, and Shapes (B 221 - 49 T), and

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* Presented at the Fifty-fourth Annual Meeting of the Society, June 18-22, 1951.

[1] 1950 Supplement to 1949 Book of ASTM Standards, Part 2.

Aluminum Bars for Electrical Purposes (Bus Bars) (B 236 – 48 T).

The committee has also recommended Specifications B 236 to Sectional Committee C-7 for approval as American Standard.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II on Sand and Permanent Mold Castings has completed specification revisions dealings with alloy compositions and is working on a comprehensive table setting forth a realistic evaluation of the relations between the various alloys in various properties. This work is being coordinated closely with that of the American Foundrymen's Society.

Subcommittee VII is preparing a Recommended Practice for the Codification of Light Metals and Alloys.

Subcommittee VIII has obtained all of the samples for the corrosion program and samples are ready for exposure.

This report has been submitted to letter ballot of the committee, which consists of 76 members; 60 members returned their ballots, of whom 53 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

I. V. WILLIAMS,
Chairman.

R. B. SMITH,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee B-7 presented to the Society through the Administrative Committee on Standards the following recommendations:

Revision of Tentative Specifications for:

Magnesium-Base Alloys in Ingot Form for Sand Castings, Die Castings and Permanent Mold Castings (B 93 – 49 T), and
Aluminum-Base Alloys in Ingot Form for Sand Castings, Die Castings and Permanent Mold Castings (B 179 – 50 T).

Tentative Revision of Standard Specifications for:

Magnesium Ingot and Stick for Remelting (B 92 – 45).

These recommendations were accepted by the Standards Committee on September 12, 1951, and the revised tentative specifications and the tentative revision of B 92 – 45 appear in the 1951 Supplement to Book of ASTM Standards, Part 2.

On November 29, 1951, the following recommendations submitted by Committee B-7 were accepted by the Administrative Committee on Standards:

Revision of Tentative Specifications for:

Magnesium-Base Alloy Sand Castings (B 80 – 49 T),
Magnesium-Base Alloy Sheet (B 90 – 49 T), and
Magnesium-Base Alloy Permanent Mold Castings (B 199 – 49 T).

The revised tentative specifications appear in the 1951 Supplement to Book of ASTM Standards, Part 2.

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[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE B-8

ON

ELECTRODEPOSITED METALLIC COATINGS*

Committee B-8 held two meetings during the year: one on October 11, 1950, in Buffalo, N. Y., in conjunction with the Fall Congress of the Electrochemical Society and one in Cincinnati, Ohio, on March 9, 1951.

C. H. Sample has been reappointed as liaison representative to the Research Committee of the American Electroplaters' Society.

The appointment of B. C. Case as official representative of the American Electroplaters' Society on ASTM Committee B-8 has been confirmed by the Advisory Committee.

The committee now consists of 132 members, of whom 64 are classified as producers, 16 as consumers, 64 as general interest and 5 as consulting members.

RECOMMENDATIONS AFFECTING STANDARDS

The committee is presenting for publication three new tentative recommended practices and is recommending revisions in three tentatives, adoption of two tentatives as standard, and the continuation of three tentatives in their present status without revision. The standards and tentatives affected, together with the revisions recommended, are given in detail in the Appendix.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[1]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Specifications, Papers, and Definitions (J. E. Stareck, chairman) has been active throughout the year, its work resulting in the actions as given in detail in the Appendix. The subcommittee has also considered the advisability of including metric equivalents for thickness in the text of specifications under the jurisdiction of Committee B-8 and has recommended to the committee that this be done. This matter is currently under further consideration by Committee B-8.

Subcommittee II on Performance Tests (H. A. Pray, chairman) has continued exposure tests of electrodeposited lead coatings on steel and copper-nickel-chromium coatings on high-carbon steel. A progress report of Subcommittee II on the latter program, including data is appended.

Subcommittee III on Conformance Tests (B. C. Case, chairman) reports progress by each of the four active sections of Subcommittee III which are engaged in studying: thickness tests, porosity tests, mechanical property tests, and luster tests.

Subcommittee IV on Electroplating Practice (K. M. Huston, chairman) has cleared three new tentative recommended practices, prepared by working sections, during the year, and Section C is revising a fourth one on the preparation of copper and copper alloys for electroplating. An early report is expected from Section F which was appointed to conduct a survey regarding the desirability of preparing a recommended practice for the preparation of plastics for electroplating.

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* Presented at the Fifty-fourth Annual Meeting of the Society, June 18–22, 1951.

[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Renewed effort is being made to appoint sections to survey the field relative to the desirability of preparing recommended practices for the preparation of malleable and cast iron, and lead and lead alloys for electroplating.

Because of lack of interest, plans to activate a section on preparation of tin and tin alloys for electroplating have been abandoned.

Subcommittee V on Supplementary Treatments for Metallic Coatings (Marc Darrin, chairman) has been active during the year. Section B on Accelerated Tests and Section C on Performance Tests have worked closely on the program outlined to study outdoor exposure and accelerated tests on several types of chromate films applied to electrodeposited zinc on steel, and report good progress. These sections will collaborate with Subcommittee II on the exposure tests of these specimens.

Section D on Cadmium Treatments has conducted substantial preliminary work on the evaluation of supplemental chromate treatments of cadmium plate.

Section E on Phosphate Treatments reports good progress on a rather extensive testing program in which steel panels, zinc-plated, phosphate-treated and coated with a clear alkyd varnish, will be subjected to various accelerated tests as well as atmospheric exposure. Variables will be acid and cyanide zinc, and thickness of phosphate coating.

This report has been submitted to letter ballot of the committee, which consists of 99 voting members; 75 members returned their ballots, of whom 69 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

C. H. SAMPLE,
Chairman.

R. B. SALTONSTALL,
Secretary.

APPENDIX

RECOMMENDATIONS AFFECTING STANDARDS RELATING TO ELECTRODEPOSITED METALLIC COATINGS

In this Appendix are given the recommendations affecting certain standards covering electrodeposited metallic coatings, which are referred to earlier in this report. The standards appear in their present form in the 1949 Book of ASTM Standards, Part 2.

NEW TENTATIVES

Committee B-8 recommends that the following three tentative recommended practices be accepted for publication as tentative, as appended hereto:[1]

Tentative Recommended Practice for the Preparation of Zinc-Base Die Castings for Electroplating,

Tentative Recommended Practice for the Preparation of and Electroplating on Aluminum Alloys, and

Tentative Recommended Practice for the Preparation of and Electroplating on Stainless Steel.

REVISION OF TENTATIVES

The committee recommends revisions in the following three tentatives:

Tentative Specifications for Electrodeposited Coatings of Zinc on Steel (A 164 – 49 T):

Section 3.—Add the following notes at the end of this section:

NOTE 1.—See Appendix III.

NOTE 2.—The performance of a zinc coating depends largely on its thickness and the kind of environment to which it is exposed. Without proof of satisfactory correlation, accelerated tests, such as the salt spray test, cannot be relied upon to predict performance in other environments, nor will these serve as comparative measures of the corrosion protection afforded by coatings of different metals. Thus the marked superiority shown by cadmium coatings over zinc coatings of equal thickness in the standard salt spray test cannot be construed as proof that this will hold true in all atmospheric environments.

Section 8.—Add a new Paragraph (*b*) as follows and reletter the present (*b*) as (*c*):

Unless otherwise agreed upon by the manufacturer and purchaser, measurements of the thickness of coating on threaded articles, such as nuts, bolts, screws and similar fasteners with complementary threads, shall be made on the shank or other smooth surfaces as nearly adjacent to the thread as practicable.

New Appendix III.—Add a new Appendix III on "Coating Thickness on Threaded Articles with Complementary Threads" as follows:

The dimensional tolerances of most threaded articles, such as nuts, bolts, screws and similar fasteners with complementary threads, normally do not permit the application of coating thickness much greater than *Type R. S.* The limitation of coating thickness on threaded fasteners imposed by dimensional tolerances (including class of fit) should be a subject for consideration wherever practicable, both by the manufacturer and by the purchaser to prevent the application of greater coating thicknesses than are generally permissible. If heavier coatings are required for satisfactory corrosion resistance, allowances must be made in the manufacture of the threaded fasteners for the tolerances necessary for plate build-up.

Tentative Specifications for Electrodeposited Coatings of Cadmium on Steel (A 165 – 49 T):

Make the same changes in these specifications as recommended for Specifications A 164 but substitute "cadmium" for "zinc" in Note 2 of Section 3 and "*Type T.S.*" for "*Type R.S.*" in Appendix III.

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[1] These new tentatives were accepted by the Society and appear in the 1951 Supplement to Book of ASTM Standards, Part 2.

Tentative Methods of Test for Local Thickness of Electrodeposited Coatings (A 219 – 45 T):

Committee B-8 joins with Committee A-5 in recommending revisions in these methods, which are under their joint jurisdiction, as follows:

Appendix I.—Revise Sections A1, A2, A3, and A4 of Appendix I as follows:

Section A1.—Delete the present Paragraph (*a*) and add the following:

(*a*) *Nature of Test.*—The magnetic methods for estimation of thickness of coatings on metals involve instruments which measure either the attractive force between a magnet and the coated specimen or the reluctance of a flux path through the coating and the basis metal. Either the attractive force or the reluctance as measured, is a function of the thickness of the coatings. These methods may be used to measure the thickness of nonmagnetic or nickel coatings on a magnetic basis metal such as steel, the thickness of magnetic coatings on a nonmagnetic basis metal, and the thickness of the nickel and copper in a composite nickel over copper coating on steel.

In Paragraph (*b*) change "principles" to "types of apparatus." Delete the second sentence, and delete the footnotes for all three items in Paragraph (*b*). To Item (*1*) add "At this time this type is the only one suitable for measuring nickel coatings and composite nickel-over-copper coatings on steel." In Item (*3*) change "electromagnetic" to "magnetic."

Delete the present Paragraph (*c*).

Section A2.—Revise the introduction on *Factors Affecting Applicability and Accuracy of Measurement* to read as follows by the addition of the italicized words: "The following conditions *may* affect the result obtained *by a given instrument.*"

In Items (*1*), (*2*), and (*3*), change "base" to "basis." In Item (*3*) add the words "and residual magnetism." In Item (*6*) after the word "shape" add the words "and strength."

Add new Item (*10*) to read as follows: "Grain orientation in the steel basis metal."

Section A3.—Revise to read as follows:

(*a*) *Calibration.*—Each instrument, including the magnet to be used, shall be calibrated before use against suitable standards and the calibration should be checked at frequent intervals.

(*b*) Thickness standards for electrodeposited coatings on flat smooth surfaces may be purchased from the National Bureau of Standards.

(*c*) When surfaces or coatings to be tested deviate from the standards described in Section A3 (*b*), as indicated in Section A2, the standards used shall be such that errors due to the factors listed in Section A2 will be small enough to permit the accuracy requirements of Section A4 to be met. The thickness of such standards shall be determined by the miscroscopic method, or by stripping methods when applicable.

Section A4.—Revise to read as follows:

Accuracy.—The instrument, its calibration, and its operation shall be such that the coating thickness can be determined within ±5 per cent of its true thickness for single coatings and ±15 per cent for composite coatings.

Delete the note in Section A4.

ADOPTION OF TENTATIVES AS STANDARD

The committee recommends that the following two tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Specifications for Electrodeposited Coatings of Nickel and Chromium on Zinc and Zinc-Base Alloys (B 142 – 45 T), and

Tentative Recommended Practice for the Preparation of High-Carbon Steel for Electroplating (B 242 – 49 T).

TENTATIVES CONTINUED WITHOUT REVISION

Because of test programs now in progress pertaining to the following tentatives, the committee recommends that they be continued in their present tentative status without revision:

Tentative Specifications for Electrodeposited Coatings of Nickel and Chromium on Steel (A 166 – 45 T),

Tentative Specifications for Electrodeposited Coatings of Lead on Steel (B 200 – 45 T), and

Tentative Specifications for Chromate Finishes on Electrodeposited Zinc, Hot-Dipped, Galvanized, and Zinc Die-Cast Surfaces (B 201 – 49 T).

REPORT OF SUBCOMMITTEE II ON PERFORMANCE TESTS

ATMOSPHERIC EXPOSURE OF COPPER-NICKEL-CHROMIUM DEPOSITS ON HIGH-CARBON STEEL

This report is essentially a continuation of the 1949 report of Subcommittee II,[1] in which the details with respect to the plan of tests, preparation of test panels, composition of the plating and cleaning solutions, characterization of the plated panels, distribution of the panels to exposure sites, the inspection method, and the objectives of the study of the atmospheric corrosion properties of copper-nickel-chromium deposits on high-carbon steel are given. The present report should, therefore, be considered as a second part of the subcommittee's report on the program on this subject. Its purpose is to present the test results that were not available for inclusion in the 1949 subcommittee report, to summarize the inspection data for the full exposure period, and to list the more obvious conclusions resulting from the study. The tests are being amplified by a supplementary set of panels which are now on exposure and which will be described in a subsequent report.

TEST RESULTS

The rating numbers and the abbreviated description of the appearance of the panels exposed at New York, N. Y., Kure Beach, N. C., State College, Pa., Pittsburgh, Pa., and Coral Gables, Fla., are given in Table I for exposure periods of about 1.5 to 3.5 yr. Similar data for periods up to about 1.5 yr are listed in Table X of the 1949 report. The descriptive abbreviations are intended to give an idea of the type of defects which resulted in the assigned average rating numbers for panels of a given lot. They are obviously qualitative and of less significance in determining the relative merits of the various coating systems than are the rating numbers.

The rating numbers from Table X (1949 report) and Table I are summarized in Table II for all of the exposure stations, including Detroit (atmospheric plus periodic sodium chloride solution), for the total of approximately 3.5 yr exposure and also including rating numbers for the 200- and 500-hr standard salt-spray tests. In considering Table II, it should be emphasized that the rating numbers are not strictly quantitative and that differences of one or two units are not very significant. The rating numbers, however, constitute the principal basis for conclusions.

Atmospheres:

It requires only a glance at Table II to determine that there is a wide difference in the effect of different atmospheric conditions on the deterioration of plated coatings of this type. The heavy industrial atmosphere (Pittsburgh) is the most damaging. Attack by the New York atmosphere is somewhat less severe than at Pittsburgh, but much more so than at the seacost environment of Kure Beach. The less contaminated atmospheres of Coral Gables and State College result in very much milder corrosion.

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[1] *Proceedings*, Am. Soc. Testing Materials, Vol. 49, pp. 226-238 (1949).

Nickel Directly on Steel:

By comparing lot Nos. 1, 5, 10, and 10X at the different exposure periods and at various exposure sites, it is evident that the protective value of nickel, without copper undercoat, is dependent upon its thickness. The heavier nickel coatings are superior in that the rating numbers at a given site are generally higher or their rating numbers decrease more slowly with time than for the thinner coatings. A 3-mil coating appears to be better than one of 2 mils, but the difference is not so striking as that between the 1-mil and 2-mil deposits.

Nickel Over a Copper Flash:

The effect of 0.1 mil of cyanide copper under nickel plates of various thicknesses can be seen by comparing lot Nos. 1 and 2, 5 and 6, 10 and 11, and 25 and 26. For a given nickel thickness at the more severe locations, a copper flash under the nickel appears to have little or no effect. There is an indication that, at State College, the copper flash is somewhat beneficial and, at Kure Beach, somewhat detrimental. The effect is small, in any case, and probably unimportant.

Nickel Over Heavy Copper Deposits:

At most of the exposure sites, lot Nos. 3, 4, 8, 9, 15, and 16 (all coated with 0.5 mil of nickel over from 0.5 to 1.5 mils of acid copper over a cyanide copper strike) show about the same corrosion behavior. Exceptions are noted at Kure Beach and State College, particularly for the shorter exposure periods. The same comparison at a nickel thickness of 1.0 mil (lot Nos. 1, 2, 7, 13, and 14) with copper varying from 0 to 1.0 mil, indicates that the corrosion behavior is about the same regardless of copper thickness. Again, State College is an exception. At a nickel thickness of 1.5 mils (lot Nos. 5, 6, 12, and 25), with copper varying from 0 to 1.5 mils, and at a nickel thickness of 2 mils (lot Nos. 10, 11, and 14X), with copper varying from 0 to 1 mil, similar rating numbers are obtained for a given nickel thickness, whatever the thickness of the copper undercoat.

Nickel Over Buffed Copper:

No clear-cut improvement resulting from buffing the copper undercoat, prior to nickel plating, is shown by comparing the rating numbers of lot Nos. 3 with 4, 8 with 9, 13 with 14, and 15 with 16. For a given copper and nickel thickness, deterioration occurs at essentially the same rate for the companion panels at any of the exposure sites.

Nickel Over Nickel:

For a given total nickel thickness, applying the nickel in two steps with the first layer buffed, or annealed and buffed, shows a remarkably beneficial effect at Kure Beach and, for the thinner coatings, at State College (compare lot Nos. 1, 4NA and 4B; 5, 9NA; 10, 14NA, 14B; and 28 and 29NA). This beneficial effect is completely absent at the more severe locations, New York and Pittsburgh.

pH of Nickel Bath:

Comparison of the results for lot Nos. 1 and 2 with those for lots Nos. 5X and 6X, the difference being solely in the pH of the nickel plating bath, reveals no significant difference at any of the sites that can be attributed to the pH of the bath.

Semibright versus Dull Nickel:

The rating numbers for comparative lots (Nos. 10 and 28; 13 and 29) indicate that the dull nickel is slightly better at Kure Beach than the semibright nickel represented in the test. The difference is not apparent at New York, Pittsburgh, or State College.

Bright versus Dull Nickel:

Although not shown in the tabulated data, the variation from panel to panel in the case of those plated with bright nickel was very much greater than for the other deposits. Generally, the rating

TABLE I.—INSPECTION DATA.

NOTE.—^{b} = buffed; ^{ba} = buffed and annealed; ^{WA} = wetting agent present; ^{pH} = high-pH bath.
All panels include a final chromium coating 0.00001 in. thick.

NEW YORK

Lot	Steel (SAE)	Coating, Mils			2.16 Yr		2.50 Yr		3.15 Yr		3.59 Yr	
		Cu	Ni	Total	Rating	Description	Rating	Description	Rating	Description	Rating	Description
No. 1.....	1095	...	1.0	1.0	3	xpR; vsRs; seZ	1	xpR; iRs; seZ	1	xpR; sRs; sS; seZ; iSp	0	xpR; icR; iRs
No. 2.....		0.1	0.9	1.0	4	“ ; seR; vseZ	2	“ ; iscR; sRs	1	“ ; icR; iSp; iRs; sS; ieZ	0	“ ; “ ; “
No. 3.....		0.5	0.5	1.0	0	“ ; xcR; xRs	0	“ ; xcR; xRs	0	scR; xRs	0	“ ; scR; xRs
No. 4.....		0.5^{b}	0.5	1.0	0	“ ; “ ; “	0	“ ; “ ; “	0	“ ; “	0	“ ; “ ; “
No. 4NA..		...	0.5^{b} +0.5	1.0	2	“ ; sRs; iW; seZ	2	“ ; iRs; iW; seZ	1	xpR; sRs; iSp; sS; xiW; seZ	0	“ ; iRs
No. 4B....		...	0.5^{ba} +0.5	1.0	4	“ ; vsRs	3	“ ; sRs	1	“ ; “ ; “ ; “	0	“ ; “
No. 5X...		...	1.0^{pH}	1.0	3	“ ; sRs	2	“ ; iRs	1	“ ; “ ; “ ; “	0	“ ; “
No. 6X...		0.1	0.9^{pH}	1.0	2	“ ; icR; iRs; isB	2	“ ; xcR; iRs; isB	1	“ ; “ ; “ ; “ ; icR	0	“ ; icR; iRs
No. 5.....	1095	...	1.5	1.5	3	xPR; vsRs	2	xpR; iRs	1	xpR; sRs; iSp; sS	0	xpR; iRs
No. 6.....		0.1	1.4	1.5	3	“ ; “	3	“ ; scR; iRs	2	“ ; “ ; “ ; “ ; scR	0	“ ; scR; iRs
No. 7.....		0.5	1.0	1.5	1	“ ; xcR; iRs	0	“ ; xcR; xRs	0	“ ; xcR; xRs	0	“ ; xcR; xRs
No. 8.....		1.0	0.5^{WA}	1.5	0	“ ; “ ; xRs; sF	0	“ ; “ ; “	0	“ ; “ ; “ ; seF	0	“ ; “ ; “
No. 9.....		1.0^{b}	0.5	1.5	0	“ ; “ ; xRs; sF	0	“ ; “ ; “	0	“ ; “ ; “	0	“ ; “ ; “
No. 9NA..		...	1.0^{b} +0.5	1.5	4	“ ; ieZ	3	“ ; iRs; seZ	1	“ ; sRs; iSp; iS	0	“ ; iRs
No. 10.....	1095	...	2.0	2.0	4	xpR; vsez	4	ipR; iSp; sRs; iS	2	xpR; sRs; iSp; iS	0	xpR; iRs
No. 11.....		0.1	1.9	2.0	4	“ ; “	4	“ ; “ ; “ ; “ ; seZ	2	“ ; “ ; “ ; “ ; scR	0	“ ; scR; iRs
No. 12.....		0.5	1.5	2.0	3	“ ; ieZ	3	spR; “ ; “ ; “ ; xZ; iW	1	“ ; “ ; “ ; “ ; vseR; iZ	0	“ ; “ ; “
No. 13.....		1.0	1.0	2.0	1	“ ; iRs; xW; ieZ	0	“ ; xSp; xS; ieZ	0	“ ; xRs; iSp; xS; iZ	0	“ ; icR; xRs
No. 14.....		1.0^{b}	1.0	2.0	1	“ ; icR; iRs; xW; ieZ	0	xpR; icR	0	“ ; “ ; “ ; “ ; xcR	0	“ ; xcR; “
No. 14NA..		...	1.0^{b} +1.0	2.0	3	“ ; seZ	3	ipR; iSp; sRs; sS; seZ	2	“ ; sRs; iSp; iS	1	“ ; iRs
No. 14B....		...	1.0^{ba} +1.0	2.0	3	“ ; “	3	“ ; “ ; “ ; “ ; “	2	“ ; “ ; “ ; “	1	“ ; “
No. 15.....		1.5	0.5	2.0	1	“ ; icR; iRs; vseZ	0	xpR; icR; iRs; iS	0	“ ; xRs; xcR; xS	0	“ ; xcR; xRs
No. 16.....		1.5^{b}	0.5	2.0	1	“ ; “ ; “ ; “	0	“ ; “ ; “ ; “	0	“ ; “ ; “ ; “	0	“ ; “ ; “
No. 10X...	1095	...	3.0	3.0	4	xpR; vseZ	4	vspR; iSp; iS	2	xpR; sRs; iSp; iS	1	xpR; sRs
No. 14X...		1.0	2.0	3.0	4	“ ; “	4	“ ; “ ; “	1	“ ; “ ; “ ; “	0	“ ; iRs
No. 16X...		1.5	1.5	3.0	3	“ ; vsRs	3	“ ; “ ; “	1	“ ; “ ; “ ; “ ; scR	0	“ ; “
No. 25.....	9112	...	1.5	1.5	4	xpR; vsRs; vseZ	3	xpR; iRs; iS; ieZ	1	xpR; xRs; iSp; iS; scR	0	xpR; scR; xRs
No. 26.....		0.1	1.4	1.5	3	“ ; ieZ; fsB	3	“ ; “ ; “ ; “	1	“ ; “ ; “ ; “ ; icR	0	“ ; icR; “
No. 27.....		0.5	1.0	1.5	1	“ ; xcR; iRs; seZ	0	“ ; xcR; xRs	0	“ ; “ ; xcR	0	“ ; xcR; “
No. 28.....	1095	...	2.0SB	2.0	3	xpR; icR; xeF; isB	3	xpR; icR; xF	0	xpR; xRs; xcR; xeF	0	xpR; scR; iRs; xF
No. 29.....		1.0	1.0SB	2.0	2	“ ; “ ; iRs; ieZ; iW	1	ipR; icR; iRs; iS; xeZ	0	“ ; “ ; “ ; xS	0	“ ; “ ; xRs
No. 29NA..		...	1.0^{b} +1.0SB	2.0	3	“	3	“ ; “ ; “ ; “ ; ieZ	1	“ ; “ ; iSp; iS; iW	0	“ ; “

No. 30.....	1095	1.0	1.0B	2.0	2	xpR; icR; iRs; iB	1	xpR; xcR; iRs; iS	0	xpR; xRs; xcR; xS	0	xpR; xcR; xRs
No. 31.....		1.0^b	1.0B	2.0	2	“ ; “ ; “ ; “	1	“ ; “ ; “ ; xSp; xS	0	“ ; “ ; “ ; “	0	“ ; scR; “
No. 32.....		0.1	0.9 +1.0B	2.0	3	“ ; xW	3	ipR; iSp; sRs; iS; iiB; seZ	1	“ ; “ ; icR; iSp; xW	0	“ ; iRs
No. 33.....		0.1	0.9^b +1.0B	2.0	3	“ ; “	3	“ ; “ ; “ ; “ “ ; “	1	“ ; “ ; “ ; “ ; “	0	“ ; “
No. 34.....	1095 High-polish	0.1	0.9 +1.0B	2.0	3	vspR; xSp	1	vspR;xSp; sS	0	ipR; xRs; xSp	0	ipR; “

KURE BEACH

Lot	Steel (SAE)	Coating, mils			1.48 Yr		2.17 Yr		3.18 Yr		3.66 Yr	
		Cu	Ni	Total	Rating	Description	Rating	Description	Rating	Description	Rating	Description
No. 1........	1095	...	1.0	1.0	5	ipR: icR; iRs; iB	3	xpR; xcR; iRs; iB	2	xpR; scR; iRs; xF	2	scR; xRs
No. 2........		0.1	0.9	1.0	4	“ ; xcR; “ ; sB	2	“ ; “ ; “ ; “	2	“ ; “ ; “ ; “	2	“ ; “
No. 3........		0.5	0.5	1.0	0	xpR; xcR; xRs; xB	0	“ ; “ ; “ ; “	0	xcR;	0	“ ; “
No. 4........		0.5^b	0.5	1.0	1	“ ; “ ; “ ; “	0	“ ; “ ; “ ; “	0	“	0	“ ; “
No. 4NA......		...	0.5^b +0.5	1.0	9	vspR; vsRs; ieR	9	spR; scR	9	vspR; vscR	9	vscR; vsRs
No. 4B......		...	0.5^{ba} +0.5	1.0	9	spR; scR; sRs	8	“ ; “	8	spR; vscR	9	“ ; “
No. 5X......		...	1.0^{pH}	1.0	6	spR; ieR; iRs; seR; sB	3	xpR; scR; iRs; B	2	xpR; xcR; xRs; xF	2	xcR; xRs
No. 6X......		0.1	0.9^{pH}	1.0	3	ipR; xcR; iRs; iB; eF	1	“ ; “ ; xRs; eB	0	xcR; xRs; xF	1	“ ; “
No. 5........	1095	...	1.5	1.5	8	spR; scR; sRs; sSp; fsB	6	ipR; vscR; vsRs	5	ipR; scR; iRs	7	icR; iRs; fsB
No. 6........		0.1	1.4	1.5	7	“ ; “ ; “ ; iSp; eF	5	“ ; icR; “	4	xpR; xcR; iRs; iF	6	“ ; “ ; iB
No. 7........		0.5	1.0	1.5	3	xpR; icR; xRs; sB; eF	2	xpR; xcR; iRs	1	“ ; “ ; xRs	2	xcR; xRs
No. 8........		1.0	0.5^{WA}	1.5	4	ipR; icR; iRs; iSp; iB; xeF	1	“ ; “ ; “ ; seZ	0	“ ; “ ; “ ; xF	2	“ ; “
No. 9........		1.0^b	0.5	1.5	4	xpR; xcR; xRs; xSp; ieF	1	“ ; “ ; “ ; “	0	“ ; “ ; “ ; “	2	“ ; “
No. 9NA......		...	1.0^b +0.5	1.5	9	vspR; vsRs; iSp; ieF	9	vscR; seZ	9	vspR; vscR	9	vscR
No. 10........	1095	...	2.0	2.0	9	spR; vsRs; vsSp; vsB	9	spR	9	spR	9	sRs
No. 11........		0.1	1.9	2.0	9	“ ; “ ; “ ;	7	ipR; scR	7	“ ; icR; iRs	7	icR; iRs
No. 12........		0.5	1.5	2.0	9	vspR; vsRs; vsSp	6	“ ; icR; vseZ	6	“ ; “ ; “	6	“ ; “
No. 13........		1.0	1.0	2.0	8	spR; scR; sRs; vsSp	5	“ “ ; sRs; vseZ	4	“ ; xcR; xRs	4	xcR; xRs
No. 14........		1.0^b	1.0	2.0	8	ipR; scR; sRs; sSp	5	“ ; “ ; “ ; “	4	“ ; “ ; “	3	“ ; “
No. 14NA.....		...	1.0^b +1.0	2.0	9	vsSp; vsRs; vseF	9	vsRs	9	vspR; vscR	9	vscR; vsRs
No. 14B.......		...	1.0^{ba} +1.0	2.0	9	vspR; vsRs; vsSp	9	“	9	“	9	“ ; “
No. 15........		1.5	0.5	2.0	2	xpR; xcR; xRs; sSp	0	xpR; xcR; xRs	0	xpR; xcR; xRs	1	“ ; “
No. 16........		1.5^b	0.5	2.0	6	“ ; icR; iRs; xSp; ieF	3	“ ; “ ; “	3	“ ; “ ; “	3	“ ; “
No. 10x......	1095	...	3.0	3.0	9	spR; sRs; iSp	9	vspR	9	vspR	9	vscR; vsRs
No. 14X......		1.0	2.0	3.0	9	vspR; vsRs; iSp	9	“	9	“	9	vsRs
No. 16X......		1.5	1.5	3.0	9	“ ; “ ; sSp	9	“	8	spR	8	vscR; sRs

TABLE I (*Continued*)
KURE BEACH (*Continued*)

Lot	Steel (SAE)	Coating, mils			1.48 Yr		2.17 Yr		3.18 Yr		3.66 Yr	
		Cu	Ni	Total	Rating	Description	Rating	Description	Rating	Description	Rating	Description
No. 25	9112	...	1.5	1.5	8	spR; scR; sRs; sSp; seF	5	ipR; xcR	5	ipR; icR; iRs; iF	5	xcR; xRs
No. 26		0.1	1.4	1.5	8	" ; " ; " ; " ; "	6	" ; "	6	" ; " ; " ; "	8	scR; sRs; isB
No. 27		0.5	1.0	1.5	3	ipR; xcR; xRs; sSp; xB	1	xpR; xcR; xRs	0	xcR; xF	0	......
No. 28	1095	...	2.0SB	2.0	6	ipR; icR; iRs; iSp; sB; xeF	2	ipR; iRs; xxB	1	xcR; xF	0	xF
No. 29		1.0	1.0SB	2.0	6	xpR; scR; " ; " ; vsB; ieF	3	xpR; xcR; sRs	3	xpR; xcR	4	xcR; xRs; xB
No. 29NA		...	1.0^b +1.0SB	2.0	7	ipR; icR; sRs; iSp; ieF	3	" ; icR; sRs	2	" ; icR; xF	3	icR; " ; "
No. 30	1095	1.0	1.0B	2.0	7	ipR; icR; sRs; xSp; iB	2	xpR; scR; sRs	2	xpR; xcR; xF	3	xcR; xRs; xB
No. 31		1.0^b	1.0B	2.0	9	spR; scR; xRs; xSp; sB; seF	5	ipR; icR; vseZ	5	" ; icR; iRs	6	icR; iRs; xSp
No. 32		0.1	0.9 +1.0B	2.0	9	spR; vscR; sRs; xSp	9	spR; vscR	9	vspR; fiB	2	vscR; vsRs; xSp
No. 33		0.1	0.9^b +1.0B	2.0	10	xSp	9	vspR; seZ	10	xSp	2	xSp
No. 34	1095 High-polish	0.1	0.9 +1.0B	2.0	10	....	9	vseZ	10	"	10	......

STATE COLLEGE

Lot	Steel (SAE)	Coating, mils			2.14 Yr		2.49 Yr		3.16 Yr		3.61 Yr	
		Cu	Ni	Total	Rating	Description	Rating	Description	Rating	Description	Rating	Description
No. 1	1095	...	1.0	1.0	6	ipR; sRs	5	ipR; iRs	5	ipR; iRs; fsB	3	ipR; sRs; fsB
No. 2		0.1	0.9	1.0	9	vspR	9	spR	8	spR; sRs; fsB	6	spR; vsRs; fsB
No. 3		0.5	0.5	1.0	4	ipR; iRs; xsB	3	xpR; iRs; xsB	2	xpR; xcR; iRs; xsB	0	xpR; sRs; xsB
No. 4		0.5^b	0.5	1.0	5	" ; sRs; "	5	" ; " ; "	3	" ; " ; " ; "	2	" ; " ; "
No. 4NA		...	0.5^b +0.5	1.0	9	vspR	9	vspR	8	spR; sRs; vfsB	9	spR; xvsB
No. 4B		...	0.5^{ba} +0.5	1.0	9	"	9	"	9	"	10	......
No. 5X		...	1.0^{pH}	1.0	7	ipR; sRs	6	ipR; sRs	6	ipR; sRs	8	spR; vsRs
No. 6X		0.1	0.9^{pH}	1.0	7	" ; vsRs; isB	6	" ; " ; xiB	5	" ; " ; xiB	8	" ; xsB
No. 5	1095	...	1.5	1.5	8	vspR; vsRs	8	vspR; vsRs	7	spR; sRs; vfsB	9	vspR
No. 6		0.1	1.4	1.5	9	"	9	"	9	"	10	......
No. 7		0.5	1.0	1.5	9	"	9	"	8	" ; isB	9	vspR
No. 8		1.0	0.5^{WA}	1.5	7	spR; vsRs; xsB	7	spR; vsRs; xsB	6	" ; sRs; xsB; eR	8	spR; sRs; seF
No. 9		1.0^b	0.5	1.5	7	" ; " ; isB	7	" ; " ; isB	6	" ; " ; " ; "	8	" ; " ; "
No. 9NA		...	1.0^b +0.5	1.5	9	vspR	9	vspR	9	"	10	......

No. 10	1095	...	2.0	2.0	9	vspR	9	vspR	9	vspR	10	......
No. 11		0.1	1.9	2.0	9	"	9	"	9	"	10	......
No. 12		0.5	1.5	2.0	9	"	9	"	9	"	10	......
No. 13		1.0	1.0	2.0	9	"	9	"	9	"	10	......
No. 14		1.0^b	1.0	2.0	9	"	9	"	9	"	10	......
No. 14NA		...	1.0^b +1.0	2.0	9	"	9	"	9	"	10	......
No. 14B		...	1.0^{ba} +1.0	2.0	9	"	9	"	9	"	10	......
No. 15		1.5	0.5	2.0	9	"	9	"	9	" ; fsB	9	vspR; fsB
No. 16		1.5^b	0.5	2.0	9	"	9	"	9	"	9	"
No. 10X	1095	...	3.0	3.0	9	vspR	9	vspR	9	vspR	10	......
No. 14X		1.0	2.0	3.0	9	"	9	"	9	"	10	......
No. 16X		1.5	1.5	3.0	9	"	9	"	9	"	10	......
No. 25	9112	...	1.5	1.5	8	vspR; vsRs	8	spR; vsRs	8	spR; sRs	8	vspR
No. 26		0.1	1.4	1.5	9	"	9	vspR	9	vspR	10	......
No. 27		0.5	1.0	1.5	9	"	9	"	9	"	10	......
No. 28	1095	...	2.0SB	2.0	8	vspR; xeF	8	vspR; xeF	8	spR; xeF	8	sF
No. 29		1.0	1.0SB	2.0	9	"	9	"	9	vspR; seR	9	isB
No. 29NA		...	1.0^b +1.0SB	2.0	9	"	9	"	9	"	10	....
No. 30	1095	1.0	1.0B	2.0	8	spR	8	spR	8	vspR	9	sSp; seF
No. 31		1.0^b	1.0B	2.0	8	"	8	"	8	"	9	" ; "
No. 32		0.1	0.9 +1.0B	2.0	8	"	8	"	8	"	8	" ; " ; xvsW
No. 33		0.1	0.9^b +1.0B	2.0	8	" ; sW	8	" ; xsW	8	"	8	" ; " ; "
No. 34	1095 High-polish	0.1	0.9 +1.0B	2.0	9	vspR	9	vspR	8	"	9	vsS

PITTSBURGH

Lot	Steel (SAE)	Coating, mils			2.14 Yr		2.50 Yr		3.15 Yr	
		Cu	Ni	Total	Rating	Description	Rating	Description	Rating	Description
No. 1	1095	...	1.0	1.0	0	xpR; xRs; fxB; ieZ	0	xpR; xRs; fxB; ieZ	0	xpR; xRs; fxB; ieZ
No. 2		0.1	0.9	1.0	0	" ; scR; xRs; ieZ	0	" ; icR; xRs; ieZ	0	" ; icR; xRs; ieZ
No. 3		0.5	0.5	1.0	0	xcR; xRs	0	xcR; xRs	0	xcR; xRs
No. 4		0.5^b	0.5	1.0	0	" ; "	0	" ; "	0	" ; "
No. 4NA		...	0.5^b +0.5	1.0	0	xpR; xRs	0	xpR; xRs; ieZ	0	xpR; xRs; ieZ
No. 4B		...	0.5^{ba} +0.5	1.0	0	" ; "	0	" ; "	0	" ; "
No. 5X		...	1.0^{pH}	1.0	0	" ; " ; ieZ	0	" ; " ; ieZ	0	" ; " ; ieZ
No. 6X		0.1	0.9^{pH}	1.0	0	" ; icR; xRs; ieB	0	" ; xcR; xRs	0	" ; xcR; xRs

TABLE I. (*Continued*)
PITTSBURGH (*Continued*)

Lot	Steel (SAE)	Coating, mils			2.14 Yr		2.50 Yr		3.15 Yr	
		Cu	Ni	Total	Rating	Description	Rating	Description	Rating	Description
No. 5	1095	...	1.5	1.5	0	xpR; xRs; fsB; ieZ	0	xpR; xRs; fiB; ieZ	0	xpR; xRs; fiB; ieZ
No. 6		0.1	1.4	1.5	0	“ ; “ ; seZ	0	“ ; icR; xRs; seZ	0	“ ; icR; xRs; seZ
No. 7		0.5	1.0	1.5	0	“ ; icR; xRs; ieZ	0	“ ; xcR; xRs; ieZ	0	“ ; xcR; xRs; ieZ
No. 8		1.0	0.5^{WA}	1.5	0	“ ; xcR; xRs	0	“ ; “ ; “	0	“ ; “ ; “
No. 9		1.0^b	0.5	1.5	0	“ ; “ ; “	0	“ ; “ ; “	0	“ ; “ ; “
No. 9NA		...	1.0^b +0.5	1.5	1	“ ; xRs	0	“ ; xRs; ieZ	0	“ ; xRs; ieZ
No. 10	1095	...	2.0	2.0	2	xpR; xRs	0	xpR; xRs; iS	0	xpR; xRs; iS
No. 11		0.1	1.9	2.0	3	“ ; iRs	0	“ ; icR; xRs	0	“ ; icR; xRs
No. 12		0.5	1.5	2.0	2	“ ; “	0	ipR; xSp; xS	0	ipR; xSp; xS
No. 13		1.0	1.0	2.0	0	“ ; xRs	0	xpR; xRs	0	xpR; xRs
No. 14		1.0^b	1.0	2.0	0	“ ; xcR; xRs	0	“ ; xcR; xRs	0	“ ; xcR; xRs
No. 14NA		...	1.0^b +1.0	2.0	2	“ ; xRs	0	“ ; xRs; ieZ	0	“ ; xRs; ieZ
No. 14B		...	1.0^{ba} +1.0	2.0	2	“ ; “	1	ipR; xSp; sRs; xS; seZ	1	ipR; xSp; sRs; xS; seZ
No. 15		1.5	0.5	2.0	0	“ ; xcR; xRs	0	xpR; xcR; xRs	0	xpR; xcR; xRs
No. 16		1.5^b	0.5	2.0	0	“ ; “ ; “	0	“ ; “ ; “ ; ieF	0	“ ; “ ; “ ; ieF
No. 10X	1095	...	3.0	3.0	3	xpR; iRs	1	ipR; xSp; sRs; xS; seZ	1	ipR; xSp; sRs; xS; seZ
No. 14X		1.0	2.0	3.0	2	“ ; “	0	spR; xSp; xS	0	xpR; xSp; xS
No. 16X		1.5	1.5	3.0	0	“ ; xRs	0	“ ; “ ; “	0	spR; xSp; xS
No. 25	9112	...	1.5	1.5	0	xpR; xRs	0	xpR; xRs; iZ	0	xpR; xRs; iZ
No. 26		0.1	1.4	1.5	0	“ ; “ ; icR; iiB	0	“ ; “ ; xxB; icR	0	“ ; icR; xRs; xB
No. 27		0.5	1.0	1.5	0	“ ; xcR; xRs	0	“ ; icR; xRs; ieZ	0	“ ; “ ; “ ; ieZ
No. 28	1095	...	2.0SB	2.0	0	xpR; xRs; iW; xF	0	xpR; xRs; xF	0	xpR; xRs; xF
No. 29		1.0	1.0SB	2.0	0	“ ; icR; xRs	0	“ ; xcR; xRs	0	“ ; xcR; xRs
No. 29NA		...	1.0^b +1.0SB	2.0	2	“ ; xRs; iiB	0	“ ; xRs; fxB	0	“ ; xRs; fxB
No. 30	1095	1.0	1.0B	2.0	0	xpR; xcR; xRs	0	xpR; xcR; xRs	0	xpR; xcR; xRs
No. 31		1.0^b	1.0B	2.0	0	“ ; “ ; “	0	“ ; “ ; “	0	“ ; “ ; “
No. 32		0.1	0.9 +1.0B	2.0	2	“ ; xRs; fsB; xiW	0	“ ; “ ; “ ; xW	0	“ ; “ ; “ ; xW
No. 33		0.1	0.9^b +1.0B	2.0	2	“ ; “ ; xiW	0	“ ; “ ; “ ; “	0	“ ; “ ; “ ; “
No. 34	1095 High-polish	0.1	0.9 +1.0B	2.0	2	ipR; xRs	0	ipR; xSp; iRs; iS	0	ipR; xSp; iRs; iS

TABLE I. (*Continued*)
CORAL GABLES.

Lot	Steel (SAE)	Coating, mils			0.36 Yr		1.48 Yr		1.75 Yr		2.0 Yr		3.0 Yr	
		Cu	Ni	Total	Rating	Description	Rating	Description	Rating	Description	Rating	Description	Rating	Description
No. 1......	1095	...	1.0	1.0	10		6	ipR; sRs	7	spR; sRs	6	spR; sRs; seR	6	spR; sRs; seR
No. 2......		0.1	0.9	1.0	10		7	" ; "	8	" ; " ; ieR	8	" ; " ; ieR	8	" ; " ; ieR
No. 3......		0.5	0.5	1.0	10		2	xpR; iRs; xiB	3	ipR; iRs; xsB	2	xpR; iRs; ieR; xiB	2	xpR; iRs; ieR; xiB
No. 4......		0.5^{b}	0.5	1.0	10		3	" ; " ; "	7	" ; sRs; vseR	6	ipR; iRs; ieR	6	ipR; iRs; ieR
No. 4NA..		...	0.5^{b} +0.5	1.0	..	...	.		.		.		.	
No. 4B....		...	0.5^{ba} +0.5	1.0	..	...	.		.		.		.	
No. 5X....		...	1.0^{pH}	1.0	10		8	spR; fsB	8	spR; sRs; seF	8	spR; sRs; ieF; fsB	8	spR; sRs; seR; ieF; fsB
No. 6X....		0.1	0.9^{pH}	1.0	10		7	ipR; sRs; seF	7	" ; " ; seF; seB	6	ipR; iRs; ieF; fsB	6	ipR; iRs; ieF; fib
No. 5......	1095	...	1.5	1.5	10		9	vspR; vsSp	9	spR; vsRs	9	spR; sRs; vseR; vfiB	9	spR; sRs; seR; vfiB
No. 6......		0.1	1.4	1.5	10		9	" ; "	8	vspR; iRs; seF	8	vspR; iRs; seF	8	vspR; sRs; seF
No. 7......		0.5	1.0	1.5	10		8	" ; "	8	" ; sRs; seF	8	" ; sRs; seF	8	" ; " ; seF
No. 8......		1.0	0.5^{WA}	1.5	10		6	spR; vsSp; fsB	7	spR; sRs; seF; sB	6	ipR; iRs; seF; fiB	6	ipR; sRs; ieF; seR
No. 9......		1.0^{b}	0.5	1.5	10		8	" ; "	8	vspR; sRs; xeF	8	spR; sRs; xeF	8	spR; sRs; xeF
No. 9NA..		...	1.0^{b} +0.5	1.5	..	...	.		.		.		.	
No. 10.....	1095	...	2.0	2.0	10		9	vspR	9	vspR; vsRs	9	vspR; vsRs	9	vspR; vsRs
No. 11.....		0.1	1.9	2.0	10		9	"	9	" ; "	9	" ; " ; vseF	9	" ; " ; vseF
No. 12.....		0.5	1.5	2.0	10		8	spR; vsSp	9	" ; "	9	" ; "	9	" ; " ; "
No. 13.....		1.0	1.0	2.0	10		9	vspR	9	" ; " ; seF	9	" ; " ; vseF	9	" ; " ; "
No. 14.....		1.0^{b}	1.0	2.0	10		9	"	9	" ; "	9	" ; "	9	" ; "
No. 14NA.		...	1.0^{b} +1.0	2.0	..	...	.		.		.		.	
No. 14B...		...	1.0^{ba} +1.0	2.0	..	...	.		.		.		.	
No. 15.....		1.5	0.5	2.0	10		7	spR	8	ipR; iRs	7	ipR; iRs; seF	7	ipR; sRs; seF
No. 16.....		1.5^{b}	0.5	2.0	10		7	"	8	spR; sRs; seF	8	" ; sRs; seF	8	" ; " ; "
No. 10X...	1095	...	3.0	3.0	10		9	sSp	9	vspR; vsRs	9	vspR; vsRs	9	vspR; vsRs
No. 14X...		1.0	2.0	3.0	10		9	vspR; vsSp	9	" ; "	9	" ; "	9	" ; "
No. 16X...		1.5	1.5	3.0	10		9	" ; "	9	" ; "	9	" ; "	9	" ; "
No. 25.....	9112	...	1.5	1.5	10		9	vspR; vsSp	9	vspR; vsRs	9	vspR; vsRs; ieF	9	vspR; vsRs; ieF
No. 26.....		0.1	1.4	1.5	10		9	" ; "	9	" ; " ; seF	9	" ; sRs	9	" ; sRs
No. 27.....		0.5	1.0	1.5	10		7	ipR; fiB	8	spR; sRs; seF; sB	7	spR; sRs; seF; fiB	7	spR; sRs; seF; fiB
No. 28.....	1095	...	2.0SB	2.0	10		9	vspR; vsSp; xF	8	vspR; sRs; ieB	8	vspR; iRs; ieB	8	vspR; iRs; ieB; xF
No. 29.....		1.0	1.0SB	2.0	10		9	" ; " ; seF	8	" ; " ; seF	8	" ; sRs; seF	8	" ; sRs; seF
No. 29NA.		...	1.0^{b} +1.0SB	2.0	..	...	.		.		.		.	
No. 30.....	1095	1.0	1.0B	2.0	10		5	ipR; vsSp	8	spR; iRs; seF	7	ipR; iRs; seF	7	ipR; iRs; seF
No. 31.....		1.0^{b}	1.0B	2.0	10		6	" ; "	7	" ; sRs; ieF	7	spR; sRs; seF	7	spR; sRs; ieF
No. 32.....		0.1	0.9 +1.0B	2.0	10		8	spR; vsSp	9	vspR; vsRs; vseB	9	vspR; vsRs	9	vspR; vsRs; fiB
No. 33.....		0.1	0.9^{b} +1.0B	2.0	10		9	vspR	9	" ; " ; vseF	9	" ; " ; vseF	9	" ; " ; vseF
No. 34.....	1095 High-polish	0.1	0.9 +1.0B	2.0	10		9	"	9	" ; "	9	" ; "	9	" ; "

TABLE II.—RATING NUMBER SUMMARY.

Lot	Steel (SAE)	Coating Thickness, mils			Exposure Time, yr.																																								Standard Salt-Spray	
					New York									Kure Beach							State College								Pittsburgh								Coral Gables					Detroit + Salt Solution				
		Cu	Ni	Total	0.10	0.57	0.96	1.22	1.46	2.16	2.50	3.15	3.59	0.13	0.62	1.17	1.48	2.17	3.18	3.66	0.13	0.58	1.17	1.47	2.14	2.49	3.16	3.61	0.12	0.57	0.95	1.16	1.47	2.14	2.50	3.15	0.36	1.48	1.75	2.0	3.0	0.58	0.95	1.17	200 hr	500 hr
No. 1	1095	...	1.0	1.0	10	4	4	4	3	3	1	1	0	9	8	5	5	3	2	2	10	9	8	8	6	5	5	3	9	0	1	2	0	0	0	0	10	6	7	6	6	2	2	2	9	8
No. 2		0.1	0.9	1.0	10	4	4	4	3	4	2	1	0	10	8	5	4	2	2	2	10	9	10	9	9	9	8	6	10	2	1	2	0	0	0	0	10	7	8	8	8	2	2	2	10	9
No. 3		0.5	0.5	1.0	10	2	2	2	1	0	0	0	0	5	1	0	0	0	0	0	10	8	9	6	4	3	2	0	8	0	0	0	0	0	0	0	10	2	3	2	2	0	0	0	9	9
No. 4		0.5^{b}	0.5	1.0	10	2	2	2	1	0	0	0	0	8	1	0	1	0	0	0	10	9	9	8	5	5	3	2	7	0	0	0	0	0	0	0	10	3	7	6	6	0	0	0	9	9
No. 4NA		...	0.5^{b} +0.5	1.0	10	4	4	3	3	2	2	1	0	10	10	9	9	9	9	9	10	10	10	9	9	9	8	9	10	1	1	2	0	0	0	0	...	...	...	...	...	...	...	...	10	10
No. 4B		...	0.5^{ba} +0.5	1.0	10	5	5	5	4	4	3	1	0	10	9	9	9	8	8	9	10	10	10	10	9	9	9	10	10	0	1	3	0	0	0	0	...	...	...	...	...	...	...	...	10	9
																													10	0	1	3	0	0	0	0										
No. 5X		...	1.0^{pH}	1.0	10	4	4	4	3	3	2	1	0	10	9	7	6	3	2	2	10	9	9	9	7	6	6	8	10	0	1	2	0	0	0	0	10	8	8	8	8	1	1	1	10	9
No. 6X		0.1	0.9^{pH}	1.0	10	4	4	4	3	2	2	1	0	10	8	4	3	1	0	1	10	9	9	9	7	6	5	8	10	2	1	2	0	0	0	0	10	7	7	6	6	1	1	1	10	10
No. 5	1095	...	1.5	1.5	10	4	4	4	3	3	2	1	0	10	9	8	8	6	5	7	10	9	9	9	8	8	7	9	10	2	1	3	1	0	0	0	10	9	9	9	9	3	2	2	9	9
No. 6		0.1	1.4	1.5	10	5	4	5	4	3	3	2	0	10	9	8	7	5	4	6	10	10	10	10	9	9	9	10	10	3	1	3	2	0	0	0	10	9	8	8	8	3	3	2	10	10
No. 7		0.5	1.0	1.5	10	3	3	3	2	1	0	0	0	9	6	4	3	2	1	2	10	10	10	10	9	9	8	9	10	2	0	0	0	0	0	0	10	8	8	8	8	2	0	0	9	9
No. 8		1.0	0.5	1.5	10	2	2	2	1	0	0	0	0	9	5	3	4	1	0	2	10	7	9	7	7	7	6	8	7	0	0	0	0	0	0	0	10	6	7	6	6	0	0	0	9	8
No. 9		1.0^{b}	0.5	1.5	10	2	2	2	1	0	0	0	0	9	5	3	4	1	0	2	10	8	9	7	7	7	6	8	7	0	0	0	0	0	0	0	10	8	8	8	8	0	0	0	10	9
No. 9NA		...	1.0^{b} +0.5	1.5	10	4	5	4	3	4	3	1	0	10	9	9	9	9	9	9	10	10	10	10	9	9	9	10	10	4	1	3	0	1	0	0	...	...	...	...	...	...	...	...	10	10
No. 10	1095	...	2.0	2.0	10	5	5	5	4	4	4	2	0	10	9	9	9	9	9	9	10	10	10	10	9	9	9	10	10	5	2	4	3	2	0	0	10	9	9	9	9	3	3	2	10	10
No. 11		0.1	1.9	2.0	10	5	5	6	4	4	4	2	0	10	9	9	9	7	7	7	10	10	10	10	9	9	9	10	10	5	4	4	3	3	0	0	10	9	9	9	9	3	3	2	9	9
No. 12		0.5	1.5	2.0	10	5	4	5	4	3	3	1	0	10	9	8	9	6	6	6	10	9	10	10	9	9	9	10	10	4	4	4	3	2	0	0	10	8	9	9	9	3	3	2	10	9
No. 13		1.0	1.0	2.0	10	4	3	2	2	1	0	0	0	10	8	7	8	5	4	4	10	10	10	9	9	9	9	10	10	3	2	2	0	0	0	0	10	9	9	9	9	2	2	2	10	9
No. 14		1.0^{b}	1.0	2.0	10	4	3	3	2	1	0	0	0	10	9	6	8	5	4	3	10	10	10	9	9	9	9	10	10	3	0	0	0	0	0	0	10	9	9	9	9	2	0	0	10	9
No. 14NA		...	1.0^{b} +1.0	2.0	10	5	5	6	4	3	3	2	1	10	9	9	9	9	9	9	10	10	10	10	9	9	9	10	10	5	3	4	1	2	0	0	...	...	...	...	...	...	...	...	10	10
No. 14B		...	1.0^{ba} +1.0	2.0	10	5	5	6	4	3	3	2	1	10	9	9	9	9	9	9	10	10	10	10	9	9	9	10	10	5	4	4	1	2	1	1	...	...	...	...	...	...	...	...	10	9
No. 15		1.5	0.5	2.0	10	2	2	2	1	1	0	0	0	7	4	2	2	0	0	1	10	10	10	8	9	9	9	9	8	0	0	0	0	0	0	0	10	7	8	7	7	0	0	0	10	9
No. 16		1.5^{b}	0.5	2.0	10	2	2	2	1	1	0	0	0	8	5	5	6	3	3	3	10	10	10	9	9	9	9	9	8	0	0	0	0	0	0	0	10	7	8	8	8	0	0	0	10	9
No. 10X	1095	...	3.0	3.0	10	5	5	6	4	4	4	2	1	10	9	9	9	9	9	9	10	10	10	10	9	9	9	10	10	4	4	4	3	3	1	1	10	9	9	9	9	3	3	2	10	10
No. 14X		1.0	2.0	3.0	10	5	5	6	4	4	4	1	0	10	10	9	9	9	9	9	10	10	10	10	9	9	9	10	10	5	4	4	3	2	0	0	10	9	9	9	9	4	5	5	10	10
No. 16X		1.5	1.5	3.0	10	4	4	4	3	3	3	1	0	10	10	9	9	9	8	8	10	10	10	10	9	9	9	10	10	5	3	3	1	0	0	0	10	9	9	9	9	2	2	1	10	10
No. 25	9112	...	1.5	1.5	10	4	4	4	3	4	3	1	0	10	9	8	8	5	5	5	10	9	9	9	8	8	8	8	10	2	1	2	0	0	0	0	10	9	9	9	9	2	3	1	10	9
No. 26		0.1	1.4	1.5	10	4	4	6	3	3	3	1	0	10	9	9	8	6	6	8	10	10	10	10	9	9	9	10	10	3	1	2	0	0	0	0	10	9	9	9	9	2	3	2	10	9
No. 27		0.5	1.0	1.5	10	3	3	3	2	1	0	0	0	9	6	3	3	1	0	0	10	9	10	9	9	9	9	10	10	0	0	0	0	0	0	0	10	7	8	7	7	0	0	0	10	9
No. 28	1095	...	2.0SB	2.0	10	4	4	5	4	3	3	0	0	10	8	7	6	2	1	0	10	10	9	8	8	8	8	8	10	5	2	4	2	0	0	0	10	9	8	8	8	3	3	3	10	9
No. 29		1.0	1.0SB	2.0	10	3	2	3	2	2	1	0	0	10	7	7	6	3	3	4	10	8	10	9	9	9	9	9	10	0	0	0	0	0	0	0	10	9	8	8	8	2	0	0	10	10
No. 29NA		...	1.0^{b} +1.0SB	2.0	10	3	4	4	3	3	3	1	0	10	9	7	7	3	2	3	10	10	10	10	9	9	9	10	10	1	0	3	0	2	0	0	...	...	...	...	...	...	...	...	10	10
No. 30	1095	1.0	1.0B	2.0	10	3	3	4	2	2	1	0	0	9	6	6	7	2	2	3	10	10	10	8	8	8	8	9	9	1	0	1	0	0	0	0	10	5	8	7	7	2	0	0	9	8
No. 31		1.0^{b}	1.0B	2.0	10	3	3	2	2	2	1	0	0	10	7	8	9	5	5	6	10	10	10	9	8	8	8	9	9	0	0	0	0	0	0	0	10	6	7	7	7	2	0	0	10	9
No. 32		0.1	0.9 +1.0B	2.0	10	4	4	5	4	3	3	1	0	10	9	9	9	9	9	2	10	10	10	8	8	8	8	8	10	2	3	4	2	2	0	0	10	8	9	9	9	3	3	2	10	9
No. 33		0.1	0.9^{b} +1.0B	2.0	10	4	4	5	4	3	3	1	0	10	10	9	10	9	10	2	10	10	10	8	8	8	8	8	10	3	3	4	2	2	0	0	10	9	9	9	9	3	3	3	10	10
No. 34	1095 high-polish	0.1	0.9 +1.0B	2.0	10	3	3	2	3	3	1	0	0	10	10	9	10	9	10	10	10	10	10	9	9	9	8	9	10	0	2	4	0	2	0	0	10	9	9	9	9	2	3	1	10	10

numbers for those panels showing no obvious plating defects were averaged for the record. On this basis, no consistent differences are evident in corrosion behavior between bright and dull nickel for comparative lots (Nos. 13 and 30; 14 and 31). No basis for a general comparison of bright versus dull nickel is provided by the limited data.

Steel Composition:

Lot Nos. 5, 6, and 7 are identical with lot Nos. 25, 26, and 27, respectively, except for the composition of the steel base. The rates of deterioration of corresponding deposits on the two base materials are essentially the same.

Degree of Polish:

A single comparison can be made between identical plates on the same steel with the standard polish and with a polish considerably superior to that on the other lots (Nos. 33 and 34). No significant difference in corrosion behavior can be found at any of the exposure sites.

Salt Spray versus Atmospheric:

The rating numbers resulting from standard salt-spray tests of 200 and 500 hr duration are listed in Table II. It is obvious that the numbers bear little or no relationship to those resulting from atmospheric exposure, although they do indicate that, by salt-spray standards, the plated coatings are satisfactorily representative of good plating practice. It should not be construed that this lack of correlation with atmospheric results is, in any sense, a condemnation of the salt-spray test as a routine quality control procedure. It does demonstrate the impossibility of distinguishing between coating types with respect to atmospheric behavior.

CONCLUSIONS

Within the range of variables represented by the test panels and the limitations of static atmospheric exposure tests, the following conclusions result from the presently available data:

1. The type of atmosphere to which copper-nickel-chromium deposits on steel were exposed had a very large effect on the rate of their deterioration as protective and decorative coatings.
2. The thickness of the nickel plate, either directly plated on steel or as an intermediate component of a composite copper-nickel-chromium coating, was the principal determining factor in the atmospheric weathering characteristics of such coatings on steel in the wide range of atmospheres represented in the tests.
3. The copper undercoatings (a cyanide copper strike or a cyanide copper strike plus acid copper) added little to the protective value of composite copper-nickel-chromium coating on steel, and what little they did add was important only in the early stages of deterioration or in the mildest corrosive atmospheres.
4. Buffing of a copper undercoat in a composite copper-nickel-chromium deposit on steel may have had a slightly beneficial effect in the early life of such coatings or in mildly corrosive atmospheres, but the effect was minor and was not persistent.
5. Applying a nickel coating in two steps, with the first layer buffed, or annealed and buffed, was highly beneficial in a seacoast environment, but was of no benefit in industrial atmospheres.
6. The type of nickel plate (dull, semi-bright, or bright) exerted no important influence on coating life, at least within the range of plate types included in the tests.
7. Identical coating systems, within the range of the simple and composite coatings studied, behaved similarly on a high-carbon steel base and on the low-alloy, high-yield-strength steel tested.

Respectfully submitted on behalf of the subcommittee,

H. A. Pray,
Chairman.

REPORT OF COMMITTEE B-9

ON

METAL POWDERS AND METAL POWDER PRODUCTS*

This report covers the activities of Committee B-9 on Metal Powders and Metal Powder Products for the period from June, 1950, to June, 1951. During this time, one meeting was held in St. Louis, Mo., on February 23, 1951.

During the year it became necessary for Alexander Squire to resign from the committee because of a change in his field of activity. F. V. Lenel was appointed by the Advisory Committee as the new secretary of the committee to serve until the next election of officers. Similarly, J. F. Kuzmick and F. P. Peters were replaced on the Advisory Committee by J. R. Boettler and E. H. Kelton.

NEW TENTATIVE

The committee recommends to the Society the publication as tentative of the Specifications for Sintered Metal Powder Structural Parts from Bronze, as appended hereto.[1] These specifications have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.

REVISION OF TENTATIVE

Tentative Specifications for Metal Powder Sintered Bearings (Oil Impregnated) (**B 202 - 45 T**)[2].—The committee recommends the following revision of these specifications:

Table I.—In Table I on Chemical Requirements change "carbon, per cent" for Type I, Class A and Class B, from "1.50 max" to "1.75 max."

This recommendation has been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.

STANDARD TENSILE TEST BARS

As reported in the 1950 Report of Committee B-9[3] the proposed Standard Tension Test Bars for Metal Powder Products had been submitted to letter ballot of the committee. Five negative votes were cast on the Round, Machined Tension Test Bar because of the erroneous impression that a choice between the Machined and Unmachined Bars was expected. During the February 23 meeting of the Committee, this matter was clarified and the committee unanimously approved the transmittal of the two test bars to Committee E-1 for issuance as ASTM standards.

ACTIVITIES OF ADVISORY COMMITTEE

The Advisory Committee held two meetings during the year to act on various problems connected with the administration of the committee. The Advisory Committee devoted attention to possible activity in the field of radio cores. A study group, consisting of J. R. Boettler, chairman, E. H. Kelton and R. P. Seelig was appointed and reported that, for the present, Committee B-9 should keep in touch with the field in anticipation of future possible standardizing activity.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Nomenclature and Technical Data (F. N. Rhines, chairman).—Several new terms are under consideration for possible inclusion in the glossary. The subcommittee is contemplating the drafting of technical data sheets on all materials covered by specifications sponsored by Committee B-9. The subcommittee is also considering the issuance of recommended shop practices for brazing, electroplating, etc., of sintered metal powder products.

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* Presented at the Fifty-fourth Annual Meeting of the Society, June 18-22, 1951.

[1] The new tentative was accepted by the Society and appears in the 1951 Supplement to Book of ASTM Standards, Part 2.

[2] 1949 Book of ASTM Standards, Part 2.

[3] *Proceedings*, Am. Soc. Testing Mats., Vol. 50, p. 185. (1950).

Subcommittee II on Metal Powders (J. J. Cordiano, chairman).—The subcommittee has voted to submit the following tentative methods to Committee E-3 which has jurisdiction over all chemical analytical methods:

Proposed Tentative Method of:
Chemical Analysis of Metal Powders.

Proposed Tentative Method for Determination of:
Hydrogen Loss in Metal Powders,
Iron Content of Iron Powders, and
Insoluble Matter in Iron and Copper Powders.

The subcommittee also voted to submit to Committee E-1 on Methods of Testing a Tentative Method for Sub-Sieve Analysis of Granular Metal Powders by Air Classification. A proposed Tentative Method for the Determination of Compressibility of Metal Powders has been prepared. Prior to submitting this method to letter ballot, it was decided to try it in a number of the members' laboratories.

Subcommittee III on Metal Powder Products (R. P. Koehring, chairman).—Section A on Bearings (D. S. Urquhart, chairman) held one meeting during the year. A table of recommended running clearances and press fits for metal powder sintered bearings has been drawn up and submitted to letter ballot of the section. It is currently undergoing modification and will be subject to a further ballot. The section has accumulated the known size lists for sintered bearings and is endeavoring to arrive at a standard size list suitable for issuance by ASTM.

Section B (C. G. Johnson, chairman) has held one meeting during the year. Tentative Specifications for Sintered Metal Powder Structural Parts from Bronze have been drafted, submitted to section and committee ballot, and have received favorable votes; the specifications are appended to this report.[1] Specifications for Sintered Metal Structural Parts from Brass are in the process of being drafted. The committee members are considering drafts of specifications for copper-impregnated structural parts from iron and for porous filter materials made from metal powders.

Section C (J. C. Redmond, chairman) held one meeting during the year, but various subgroups were active throughout the year. The subgroup on Metallographic Examination for Porosity has developed a method and standards for porosity which have been checked by several laboratories with satisfactory results. Proposed tentative specifications have been prepared for action by the section. A newly constituted group on hardness started a program of cooperative tests looking towards the preparation of a Tentative Method for Rockwell Hardness Testing of Cemented Carbides. The group on Transverse Rupture Testing is preparing a tentative method. A group was appointed to gather methods for specific gravity determination and to prepare a tentative method. Further work on the development of standard grade designations was tabled.

This report has been submitted to letter ballot of the committee, which consists of 54 members; 31 returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

W. A. REICH,
Chairman.

F. V. LENEL,
Secretary.

REPORT OF JOINT COMMITTEE

ON

EFFECT OF TEMPERATURE ON THE PROPERTIES OF METALS*

During the year, the ASTM-ASME Joint Committee on Effect of Temperature on the Properties of Metals has made notable progress in carrying out certain phases of its program. Several important publications have appeared under the committee's auspices, and others are imminent; many significant technical papers were sponsored; and in its research programs of procuring latest information on the properties and testing of materials for high- and low-temperature service, significant advances were made in the work already under way, and much new work is planned.

To underwrite several extensive research projects, the committee has undertaken to raise $85,000 to cover the cost of the projects and to have a small reservoir of funds to aid with future expected research.

Meetings:

The committee and most of its panels and subcommittees met during the ASTM Annual Meeting in Atlantic City the week of June 26, and also during the ASME annual meeting in New York City the week of November 26. The excellent attendance at these meetings and the active discussions are indicative of the intense interest which continues in the work.

A series of meetings will be held during the ASTM Annual Meeting in Atlantic City the week of June 18, 1951.

Publications and Technical Papers:

A most important contribution of the committee down through the years has been to sponsor technical symposiums and individual papers and reports providing up-to-date knowledge on the properties of metals at both high and low temperatures. This current committee year was a notable one in this respect. Five full sessions at the ASTM 1950 Annual Meeting were devoted to papers on the effect of temperature, with several technical papers at other sessions. The Symposium on the Nature, Occurrence, and Effects of Sigma Phase with eleven technical papers was issued as a separate technical publication by ASTM, STP No. 110, and likewise the Symposium on Corrosion of Materials at Elevated Temperatures with its eight technical papers was issued as STP No. 108.

It is expected there will be issued in the near future an abstract of the discussions presented in the round table on heavy forgings which was sponsored by the Joint Committee's Gas Turbine Panel.

Other individual technical papers have been published in the 1950 ASTM *Proceedings.*

In technical sessions at the 1950 ASME Annual Meeting the following papers were presented under the auspices of the Joint Committee, the last-named paper being under the joint auspices of the committee and the ASME Fuels and Power Divisions:

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* Presented at the Fifty-fourth Annual Meeting of the Society, June 18–22, 1951.

An Introduction to Arc-Cast Molybdenum and Its Alloys—J. L. Ham, Climax Molybdenum Co. of Michigan

Experience With Austenitic Steels in High-Temperature Service in Petroleum Industry—M. E. Holmberg, Phillips Petroleum Co.

An Investigation of the Role of Aluminum in the Graphitization of Plain-Carbon Steel—A. M. Hall and E. E. Fletcher, Battelle Memorial Institute

Nickel-Chromium-Molybdenum Steel Valve Casting After 50,000 Hours of Service at 900 F—T. N. Armstrong and R. J. Greene, International Nickel Co., Inc.

The Creep and Stress-Rupture Testing of Steam Boiler Materials—J. B. Romer, Babcock & Wilcox Co., and H. D. Newell, The Babcock & Wilcox Tube Co.

The General Tensional Relaxation Properties of a Bolting Steel—D. N. Frey, University of Michigan

Problems Encountered in Burning Heavy Fuel Oil as Related to Attack of Metals at High and Low Temperatures and the Fouling of Tube Banks—E. F. Tibbetts, The Lummus Co., O. L. Wood, Jr., General Electric Co., D. Douglass, Hartford Electric Light Co., and V. F. Estcourt, Pacific Gas & Electric Co.

These papers will be published in the 1951 ASME *Transactions.*

Thus, under the auspices of the committee there were presented during 1950 33 technical papers exclusive of the round-table discussion and report.

Reference is made in the report of the Data and Publications Panel to a significant new series of compilations of data, which continue the type of information made available in ASTM Special Technical Publication No. 100, by Miller and Heger. The next book in the series is on austenitic steels and is now being prepared for printing.[1]

Finances:

Committee funds in the hands of the ASME, which acts as custodian for the two sponsors, amounted on March 5, 1951, to $6405.76. Of these funds there is an obligated balance of $1779.37 to the Project 18 at Battelle involving manufacturing variables; $1616.48 is obligated to the Data and Publications Panel for work at Battelle in compiling and comparing data, and $3615.12 is earmarked for Project 29 at Battelle for graphitization studies. Thus, the obligated balance totals $6010.97, leaving a free balance of $394.79.

Future Funds:

The Finance Committee, under the chairmanship of N. L. Mochel, has prepared a comprehensive brochure and is conducting an active program to raise $85,000. Of this amount, $65,000 is estimated necessary to cover four formal projects described later in the respective panel reports. Some of the balance will be necessary to complete current work, and the remainder will be held by the custodian to underwrite expected future research.

Since its organization in 1925, the committee has undertaken only four fund-raising campaigns, to which industry and the engineering foundation have subscribed over $60,000. It is this modest sum, together with the contributions in kind—such as donated services, materials, and equipment, aggregating many-fold the actual cash value—that has enabled the committee to sponsor such a tremendous volume of research.

Standards:

At the present time the committee has three standards under its jurisdiction, as follows:

Recommended Practices for:

Short-Time Elevated-Temperature Tension Tests of Metallic Materials (E 21 - 43),

Conducting Long-Time High-Temperature Tension Tests of Metallic Materials (E 22 - 41), and

Rupture Tension Tests of Metallic Material (E 85 -50 T).

Other recommended practices and testing procedures are being studied.

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[1] This has been published as STP No. 124, prepared by Simmons and Cross.

Papers and Publications:

At the 1951 ASTM Annual Meeting, the Session on Effect of Temperature on Metals, not formally sponsored by the Joint Committee, includes the following technical contributions:

Creep-Rupture of Several Sheet Steels—G. V. Smith, E. J. Dulis, and E. G. Houston, U. S. Steel Corp.

Fabrication of Chromium-Molybdenum Steel Piping for 1000–1050 F Central Station Service—R. W. Emerson, Pittsburgh Piping and Equipment Co.

Microstructural Instability of Steels for Elevated Temperature Service—G. V. Smith, W. B. Seens, H. S. Link, and P. R. Melenock, U. S. Steel Co.

Notch-Toughness of Four Alloy Steels at Low Temperatures—W. B. Seens, W. L. Jensen, U. S. Steel Co., and O. O. Miller, International Nickel Co.

Apparatus for Low-Temperature Tension Tests of Metals—R. J. Mosborg, University of Illinois

Notch Toughness of Fully Hardened and Tempered Low Alloy Steels—R. L. Rickett and J. M. Hodge, U. S. Steel Co.

Research Projects:

Descriptive notes of the formal research projects now under way, and those which are to be undertaken, appear later in this report under the respective panels or in appended reports. Meanwhile, there are currently in progress numerous research activities being carried on by members of the committee, which involve no expenditure of committee funds. A list of the projects in these three categories follows:

I. *Research Projects Covered by Formal Agreements:*
   1. Graphitization (part of Project 29).
   2. Effect of Variables on Manufacture (Project 18).
   3. High-Temperature Data Compilations.

II. *Research Work to be Undertaken Requiring Committee Funds:*
   1. Statistical Evaluation of Creep-Rupture Properties of Certain Sheet Materials (Aviation Panel).
   2. Effect of Stress Concentration on Fatigue of Metals at Elevated Temperatures (Gas Turbine Panel).
   3. Collection, Compilation and Publication of High-Temperature Data (Data and Publications Panel).
   4. Elevated Temperature Properties of Cast Iron (Steam Power Panel, in cooperation with Committee A-3).

III. *Research Projects Being Carried on Cooperatively Requiring No Committee Funds:*
   1. Exploratory Investigation of High-Temperature Sheet Materials for Aircraft Applications.
   2. Compilation of Experience With Metals Above 1500 F.
   3. Behavior and Application of Metals at Low Temperatures.
   4. Relative Merits of 18 per cent Chromium, 8 per cent Nickel and Stabilized Grades of Austenitic Steels.
   5. Necessity for Preheating and Stress Relieving Ferritic Steels Welded with Austenitic Rods.
   6. Cracking at Welds of 12 per cent Chromium Liners in Pressure Vessels.
   7. Experience With Corrosion by Vanadium Compounds.
   8. Effect of Stress Concentration Factors on Rupture Life.
   9. Effect of Surrounding Atmosphere on Creep of Metals.
   10. Spread in High-Temperature Properties of Grade B Pipe to ASTM A 106, and in Plate to ASTM A 212.

Personnel:

During the past year the committee suffered the loss through death of Mr. F. E. Bash, Driver-Harris Co. He had taken an active and constructive part in the work for many years, and the committee records its appreciation of his loyal service.

Two new members were elected to the committee as follows: M. S. Northup, representing the Standard Oil Development Co., and W. H. Rowand of Babcock & Wilcox Co.

As now constituted, the main committee has 27 members, but including the panels, there are well over 100 men active in the work.

NOTES ON THE ACTIVITIES OF CERTAIN PANELS AND RESEARCH PROJECTS

Aviation Panel (*Leo Schapiro, chairman*):

Many of the materials necessary to carry out Research Project AP1A covering the Statistical Evaluation of Creep Rupture Properties of Certain Sheet Materials, have been procured. Alloys of Type 321, 347 Cb-Ta, N-155 Cb-Ta, and Inconel have been assembled. Companies cooperating in furnishing this material to the Panel gratuitously include: Carnegie-Illinois Steel Corp., Crucible Steel Company of America, Eastern Stainless Steel Corp., Haynes Stellite Co., and The International Nickel Co., Inc. In addition to these alloys, aluminum-clad low-carbon steel is to be included.

The study will include constant load-to-rupture tests of 450 rupture specimens with full-time extension curves reported for each. Another project involving sheet materials for aircraft applications includes exploratory investigations, including tension, fatigue, thermal shock, rupture and creep tests, all of which are under way with several companies cooperating.

The Panel is giving thought to the standardization of tension test bars of precision cast alloys. This is desirable because the size, shape, and gating arrangement may have a pronounced effect on the tensile properties. Proposed rupture and creep test specimens were considered at the last meeting of the committee, and interested parties are being contacted to determine the extent of interest.

Representatives of engine and airframe builders interests are developing statements of their interest and viewpoint on a proposed project which concerns the properties of aluminum and magnesium alloys up to 350 F.

Gas Turbine Panel (*C. T. Evans, Jr., chairman*):

As soon as funds are available this Panel will push the formal research project involving the effect of stress concentration on the fatigue of metals at elevated temperatures. This program essentially will supplement another project carried on by the National Advisory Committee on Aeronautics, and the combined results will be of considerable value to the engineer, since they will cover problems of stress concentration.

The committee is interested in the effect of intermittent high-temperature fatigue at very high frequencies. Some current research is under way in the Armed Services, and the committee expects to follow this work.

The Panel always devotes a portion of its meeting to a round-table on current problems. From discussion at the December meeting it is evident that the matter of corrosion of gas turbines from burning fuel oil has been very troublesome in Europe. An important paper on the subject of corrosion as prepared by the Panel chairman is part of the recently published symposium (ASTM STP 108) noted earlier in this report. The Panel is much interested in brittle fractures resulting from welding, and in blade fastenings from thermal cycling. The Panel plans to continue its consideration of brittle behavior and include precipitation hardening types of super alloys.

Petroleum and Chemical Panel (*C. L. Clark, chairman*):

This panel sponsored two of the contributions presented at the ASME Annual Meeting, noted earlier in this report, by Messrs. Tibbetts, Wood, Douglass, and Estcourt; and by Holmberg. Several research projects under way in this group were listed in the 1950 report. They include such problems as experience with metals above 1500 F; critical review and correlation of present low-temperature testing procedures with service results; also the relative merits of various types of austenitic steels; the necessity for preheating and stress relieving ferritic steels welded with austenitic rods; and the cracking of welds in pressure vessels.

One project may have much intensified activity because of recent developments, namely, graphitization in the petroleum and chemical industry.

The Panel is cooperating closely with the ASME Boiler Code Subgroup on Stress Allowances for Ferrous Materials to determine the high-temperature properties of carbon steels which involves a study of the possible spread in properties of Grade B Pipe, ASTM Specifications A 106, and in plates covered by ASTM Specifications A 212. This cooperative work is being expedited.

Data and Publications Panel (*R. F. Miller, chairman*):

This Panel is pushing its work to compile and publish high-temperature data on metals and alloys used at elevated temperatures. Many Code groups and others have indicated their urgent need of authoritative information. Because of this situation the committee sponsored the publication in the spring of 1950 of the Miller-Heger Report (ASTM Special Technical Publication No. 100) constituting a report on the Strength of Wrought Steels at Elevated Temperatures.

After careful consideration of the best way of providing up-to-date data, the Panel concluded that it would elicit from all known sources the data on various families of steels and alloys and publish these data more promptly than would be possible if all of the materials were covered in one survey. Since there seemed to be more urgent need for information on the austenitic alloys (the chromium-nickel type) these were covered in the first survey.

The preparation of the almost 100 charts providing data for the austenitic alloys is nearing completion and these, together with the supporting tables of data, will be published by ASTM during the early summer. This publication should be of wide-spread interest and of considerable help to the various groups who need the latest information on these austenitic alloys. The book will show rupture strengths in 100, 1000, 10,000 and 100,000 hr, and creep strengths for creep rates of 1 per cent in 10,000 and in 100,000 hr. Data will also be given for yield strength, tensile strength, elongation, and reduction in area, in all ten summary sheets and tables for each material.

The Panel was very pleased with the excellent returns from this first request for data and feels that the fine cooperation shown augurs well for its comprehensive program.

Careful consideration has been given to the families of alloys which should be covered in the next survey and, after consultation with the Steam Power Panel where the opinions of the utilities and code people were coordinated, it appeared best service would be rendered industry and others concerned if several of the so-called super alloys and a number of the ferritic chromium-molybdenum and chromium-molybdenum-vanadium steels were covered. Data on the following super alloys are being requested in the survey now under way: 16-26-6, 19-9 DL, S-590, S-816, N-155, Vitallium (HS-21), X-40 (HS-31), Inconel "X", L-605 (HS-25), Refractalloy 26, and Discalloy, including modifications, substitutes, and improvements.

General Research Panel (*A. J. Herzig, chairman*):

The meetings of the General Research Panel frequently are a forum for the presentation of informal discussions of work in the field of high-temperature metallurgy. Thus, for example, at its last meeting a representative of the Office of Naval Research gave a résumé of the work of this group in the field of high-temperature research. This Panel has a project involving the effects of stress concentration factors at elevated temperature; a second progress report of the work being carried out at Westinghouse Electric Corp. has been received. Test data have been compiled on two of the several materials being studied. One is a 12 per cent chromium, 3 per cent tungsten steel; the other, a super alloy designated Alloy B. Preliminary data on the chromium-tungsten material would indicate that the presence of a notch is not necessarily a detrimental factor, at least at the stress level used in the test, but more data are needed to confirm this definitely.

Progress continues on the research project covering influence of the surrounding atmosphere on the creep and fracture of metals at elevated temperatures. It is expected there will be a progress report from the International Nickel Co. on this work in the near future.

Steam Power Panel (*N. L. Mochel, chairman*):

Two notable papers presented at the 1950 ASTM Annual Meeting and appearing in the 1950 ASTM *Proceedings* were developed at the instigation of this Panel. Any one concerned with the subjects covered should find the information and data of much interest and help. These papers cover: Welds Between Dissimilar Alloys in Full-Size Steam Piping, by R. U. Blaser, F. Eberle, and J. T. Tucker, Jr., The Babcock & Wilcox Co.; and Some Considerations in the Joining of Dissimilar Metals for High-Temperature High-Pressure Service, by O. R. Carpenter, N. C. Jessen, J. L. Oberg, and R. D. Wylie, The Babcock & Wilcox Co.

Elevated Temperature Properties of Cast Iron.—In cooperation with ASTM Committee A-3 on Cast Iron, the Joint Committee on Effect of Temperature on the Properties of Metals is sponsoring a research project to provide authoritative information on the elevated temperature properties of cast iron, and since this work seemed to fall nearer to the scope and interest of the Steam Power Panel than other groups in the Joint Committee, the sponsorship and supervision were assigned to this Panel, which will act jointly with representatives from ASTM Committee A-3.

Committee A-3 has developed a number of widely used specifications and tests covering cast iron, including the Specifications for Gray Iron Castings A 48 – 48. These have established grades of cast iron according to strength properties, and the three higher grades, 40, 50, and 60, are to be included in this research project on elevated temperature properties. Committee A-3 has been anxious for some time to procure data on cast iron and to evaluate its potential uses at higher temperatures. As a result of discussions at meetings of the respective groups and joint consideration by representatives of the Joint Committee and Committee A-3, the current program was agreed on. It will determine for grades 40, 50, and 60, creep strength, short-time tensile properties, and rate of graphitization growth; and the studies will also involve oxidation, effect of steam and effect of repeated applications of stress at various temperatures up to 1600 F in some cases. The work at the present stage will not include studies of nodular iron.

Effect of Manufacturing Variables on the Creep Properties of Steel—Project 18 (H. C. Cross, chairman):

A report on the current status of this Project appears in Appendix I.[1]

Stability of Steels as Affected by Temperature—Project 29 (J. J. Kanter, chairman):

A report on this project, which is directly concerned with graphitization, appears in Appendix II.[2]

The officers of the Joint Committee are aware of the splendid cooperation which the representatives of industry and Government accord the committee and its work, many of these men serving on the main committee and its panels. To these men and others cooperating in the work and to the Headquarters Staffs of the two sponsor societies, the officers extend their appreciation.

The committee's record of achievements rests basically on the excellent cooperation on the part of the members, the sponsors, those carrying out formal research projects for the committee, and the many others who contribute time, effort and data.

Respectfully submitted on behalf of the committee,

ERNEST L. ROBINSON,
Chairman.

F. B. FOLEY,
Vice-Chairman.

H. C. CROSS,
Secretary.

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[1] See p. 223.

[2] See p. 230.

APPENDIX I

STUDY OF EFFECTS OF VARIABLES ON THE CREEP RESISTANCE OF STEELS

(PROJECT NO. 18)

PREPARED BY H. C. CROSS AND J. A. VANECHO

FOURTH REPORT COVERING A SPONSORED RESEARCH PROJECT CARRIED OUT AT BATTELLE MEMORIAL INSTITUTE

SYNOPSIS

The test program reported was intended to determine whether a relationship between silicon and aluminum additions might be used to control the creep resistance of fine-grained carbon steels. Higher than normal silicon contents did not produce good creep properties in AISI 1015 aluminum-treated steels. High aluminum residuals in these steels may have been responsible for the observed effects, since Bardgett has indicated that creep properties may be correlated with residual aluminum.

Three previous reports have been presented to the sponsor societies on this subject (**1**, **2**, **3**).[1]

The first report in 1938, described the effect of different grain sizes produced by heat treatment on the creep resistance of carbon steels with different aluminum additions. It was suggested, however, that grain size was not believed to be the only variable affecting creep resistance, and that small variations in composition and other manufacturing variables might be significant.

The second and third reports, in 1940 and 1944, clearly indicated the beneficial effect of silicon on the creep resistance of carbon steels. The creep resistance of a silicon-killed steel was not changed much by a change in austenite grain size or cooling rate from the coarsening temperature. The earlier belief that grain size alone was not responsible for the observed differences in creep resistance was confirmed. This was clearly shown by the tests on steels with high aluminum additions, which showed poorer creep resistance when slow-cooled from the coarsening temperature as compared with the creep resistance of an air-cooled material.

The work by Glen (**6**) and the discussion of Glen's paper by Bardgett (**7**) confirmed the beneficial effect of siliconon the creep resistance of carbon steels with aluminum additions of up to 2 lb per ton.

The recent paper on creep properties of low-carbon steels by Smith and Dulis (**4**) indicated that the steels with the best creep resistance had silicon-residual aluminum ratios between 25 and 50.

Mr. W. Bardgett of the United Steels Co, Sheffield, England, stated in a private communication that the creep rate of carbon steels correlated with their soluble aluminum contents. Steels with 0.015 per cent or less soluble aluminum had lower creep rates than those with higher residuals.

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[1] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 229.

Previous work on this project has shown that 1 lb of aluminum per ton did not adversely affect the creep resistance of a silicon-killed steel in the fine-grained condition, while 2 lb of aluminum per ton did.

This work and the work of others suggested that the relationship between silicon and aluminum additions might be used to control the creep resistance of carbon steels in the fine-grained condition. The assumption was that a somewhat higher than normal silicon content might produce good creep resistance in an aluminum-treated fine-grained steel.

STEELS TESTED

Since the previous tests were made on carbon steels containing 0.16 per cent silicon, it was desired to obtain steels containing approximately 0.30 per cent silicon, with aluminum additions of zero, 1 to 1.5, and 2.5 lb per ton. Steels of the desired higher silicon content were made and supplied gratis by the United States Steel Co in the form of 1-in diameter hot-rolled bar stock. Four special steels with the desired higher silicon contents and the range of aluminum additions were made by mold additions to four ingots of a regular open-hearth heat of AISI 1015 steel. The ladle chemical analysis of the base heat No. 23T163 was as follows:

Carbon, per cent	0.14
Manganese, per cent	0.44
Phosphorus, per cent	0.015
Sulfur, per cent	0.024
Silicon, per cent	0.24
Nickel, per cent	0.07
Chromium, per cent	0.05
Molybdenum, per cent	0.02
Copper, per cent	0.02
Aluminum, lb per ton added (Ladle)	0.35 (coarse-grain practice)

The four special ingots were treated with 75 per cent ferrosilicon and aluminum shot to meet the desired chemical analysis. The required amounts of ferrosilicon and aluminum for each ingot were distributed equally into a number of paper bags. One bag was thrown into the mold at the start of the pour, and the remaining bags were added at regular intervals so that the last bag was added when the mold was approximately three-fourths filled. The amounts of the additions and the number of bags are given below:

Ingot	75 per cent Fe Si, lb	Aluminum shot, lb	Number of Bags
AS-1	15.8	0	10
AS-2	15.8	3.7	10
AS-3	15.8	8.8	10
AS-4	40.3	8.8	16

The chemical analyses (made by United States Steel Co) for the special ingots are given below:

Ingot	Carbon, per cent	Manganese, per cent	Silicon, per cent	Aluminum, lb added per ton	Aluminum, per cent	
					Soluble	Insoluble
AS-1	0.14	0.45	0.40	0.35	0.011	0.029
AS-2	0.15	0.47	0.39	1.20	0.047	0.028
AS-3	0.15	0.47	0.39	2.40	0.087	0.018
AS-4	0.15	0.45	0.58	2.40	0.088	0.015

The four ingots were rolled to 2½-in. square bars on a primary mill, and subsequently one billet from the top portion of each ingot was hot rolled into 1-in round bars.

The silicon content of ingots AS-1, -2, and -3 are somewhat higher than intended. Ingot AS-4 was made with extra high silicon so that such a steel would be available if needed.

The soluble aluminum contents of these steels were much higher than desired or expected. The aluminum contents reported are open to some question since the indicated aluminum recoveries were exceptionally high. Carbon steels with lower silicon contents normally have much lower soluble aluminum contents. For example, in the third progress report (3), silicon-aluminum killed steel 3194 (0.16 per cent Si, 1 lb aluminum per ton) had a soluble aluminum content of only 0.001 per cent, and aluminum-killed steel 5229 with 2 lb aluminum per ton added had 0.014 per cent soluble aluminum.

In the paper by Smith and Dulis (4), Si-Al-Ti and Si-Al killed steels D and E with aluminum additions of 0.5 and 1.0 lb per ton showed aluminum residuals of only 0.004 and 0.007 per cent, while a steel F with 1.8 lb per ton addition showed a residual of 0.033 per cent. Even the Si-Al killed bessemer steels I and J with 0.18 and 0.21 per cent silicon and 3.4 and 3.0 lb of aluminum per ton added only showed residuals of 0.025 and 0.047 per cent.

Contrast these above results with the results of the special steels obtained for these tests which showed a residual aluminimum of 0.047 per cent with an aluminum addition of only 1.2 lb per ton (AS-2), and the much higher value of 0.087 per cent with an aluminum addition of 2.4 lb per ton (AS-3 and -4). Even the base heat (AS-1) with only 0.35 lb per ton added showed a residual aluminum of 0.011 per cent.

The high soluble aluminum contents of these test steels are emphasized because these are extremely important in relation to the creep resistance of these steels.

TEST RESULTS

The grain-coarsening characteristics of the four steels were determined and test bars were heat treated to produce ASTM grain sizes of No. 2 to 4 and No. 6 to 8. Figures 1 through 4 show the austenite grain sizes as determined by the heat-etching method described by Day and Austin (5).

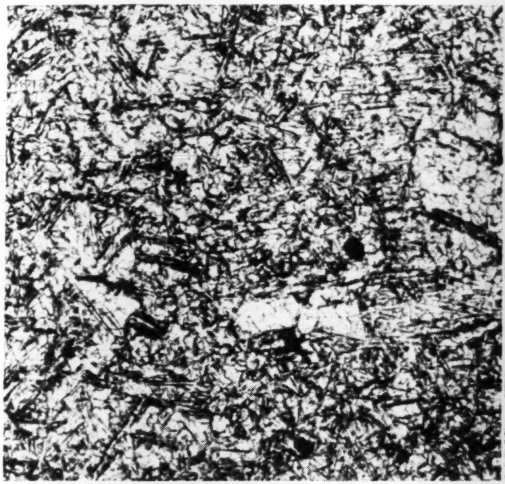

FIG. 1.—Steel AS-2, heated 1-1/2 hr at 1750 F, air-cooled. Grain size Nos. 7 to 9.

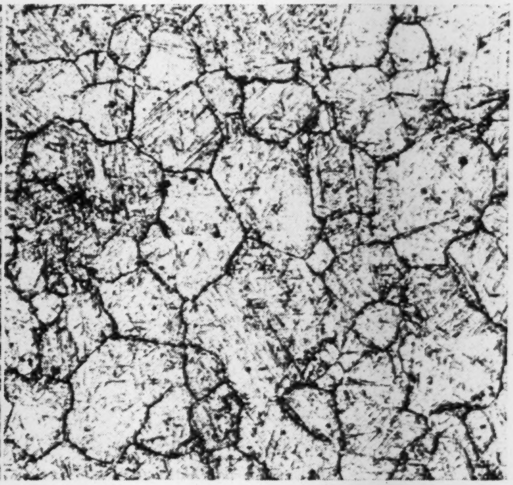

FIG. 2.—Steel AS-2, heated 1-1/2 hr at 1950 F, air-cooled. Grain size Nos. 2 to 4.

Creep tests were made at 850 F and 15,000 psi on specimens of AS-2 and AS-4 in the coarse- and fine-grained conditions, and on a specimen of AS-4 after water quenching from the grain-coarsening temperature. The creep data obtained from these tests are given in Table I, together with some previous data for comparison.

The time-deformation curves are shown in Fig. 5.

DISCUSSION OF RESULTS

The creep test of Specimen AS-2-4 (1.2 lb Al added per ton) in the fine-grained condition had a creep rate of 0.0044 per cent per hour, while Specimen AS-2-2 in the coarse-grained condition had a creep rate of 0.00070 per cent per hour. The coarse-grained specimen had the lower creep rate by a factor of six, which indicates relatively little difference in creep resistance between the coarse- and fine-grained condition. In previous tests (2), specimens of Heat 3194 (1.0 lb Al added per ton) had creep rates of 0.000126 per cent per hour and 0.000035 per cent per hour in the fine- and coarse-grained conditions, respectively. In these tests the coarse-grained specimen had the lower creep rate by a factor of 3.6.

It will be noted that the creep resistance of steel AS-2, in both the fine- and coarse-grained conditions, was considerably lower than for steel 3194. This is thought to have been caused by the much higher soluble aluminum content in steel AS-2—0.047 per cent as compared with 0.001 per cent.

Thus, with the exception of the overall lower creep properties, steel AS-2 with 0.39 Si behaved similarly to steel 3194 with 0.16 per cent Si in that a heat treatment which coarsened the austenite grain size produced an improvement in creep resistance.

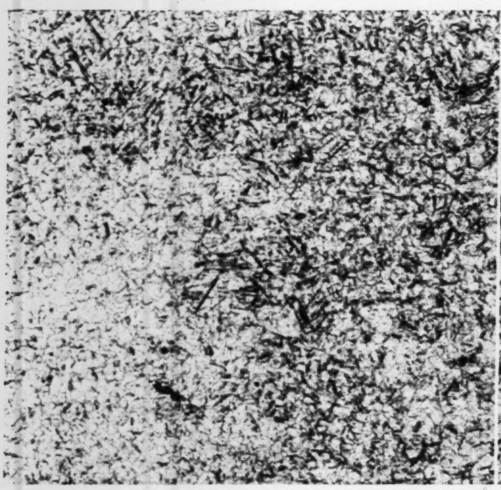

FIG. 3.—Steel AS-4, heated 6 hr at 1750 F, air-cooled. Grain size 7 to 9.

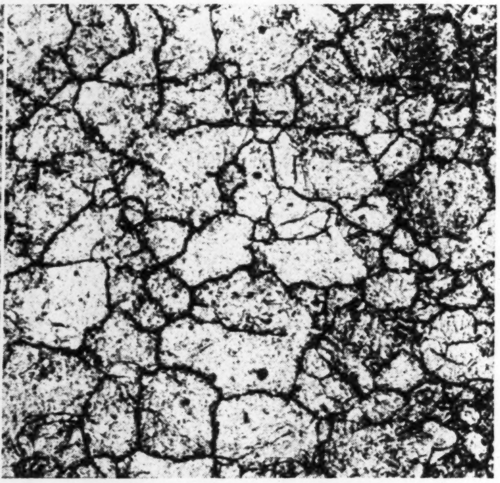

FIG. 4.—Steel AS-4, heated 1-1/2 hr at 2000 F, air-cooled. Grain size 2 to 4.

The next tests were made on steel AS-4 which contained 0.58 per cent Si and to which 2.4 lb of aluminum were added per ton of steel. The soluble aluminum content of this steel was 0.088 per cent. Fine- and coarse-grained specimens of steel AS-4 gave creep rates of 0.0045 and 0.0036 per cent per hour, respectively. For all practical purposes these rates are identical and showed no strengthening effect as a result of a coarsening treatment.

Previous work (2) showed that a steel (5280) containing 0.16 per cent Si, and to which 2.0 lb of aluminum were added per ton of steel, had a creep rate of 0.015 per cent per hour in the fine-grained condition and 0.00006 per cent per hour in the coarse-grained condition. This is an improvement of 250 to 1 on coarsening.

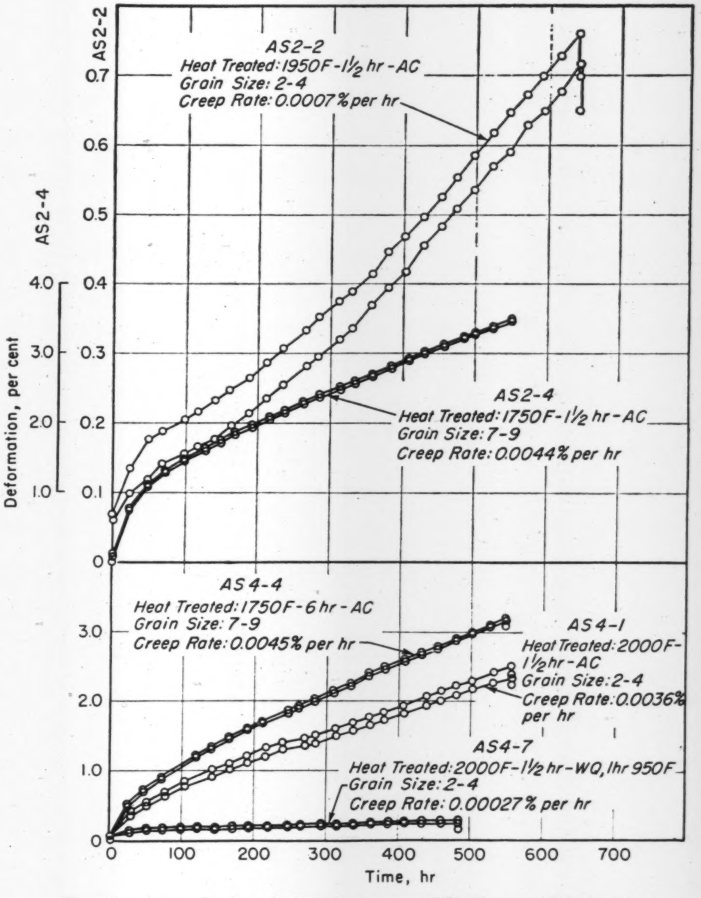

FIG. 5.—Time-Deformation Curves at 850 F and 15,000 psi.

As was stated in the introduction to this report, the purpose of these tests was to determine if some higher than normal silicon content would produce good creep resistance in a fine-grained steel. Actually, high silicon together with high aluminum has produced a steel with poor creep resistance in both the fine- and coarse-grained conditions. This is probably the result of its high residual aluminum content of 0.088 per cent.

Previous work (3) on an aluminum-killed steel had shown that the faster the cooling rate from the coarsening temperature the better the creep resistance. Table I also includes the results of creep tests on specimens of this aluminum-killed non-aging steel, 35561 (2.8 lb Al per ton), which had been either air cooled or water quenched from the grain-coarsening temperature. The air-cooled specimen had a creep rate of 0.00102 per cent per hour and the water-quenched specimen 0.00004 per cent per hour.

Thus, for this steel with an aluminum residual of 0.025 per cent, an improvement in creep rate of 25-fold was produced by rapid cooling from austenitizing temperature.

A specimen of steel AS-4 was water quenched from the coarsening temperature and tested in creep. Its creep rate was 0.00027 per cent per hour, thus showing a 13-fold improvement over the air-cooled specimen. These results are in line with the previously stated hypothesis that "at least in plain carbon steels it seems to be the absence or presence of inclusions of various types and composition whose size, mode, and degree of dispersion determine in significant manner their creep resistance" (3). The fact that the improvement for steel AS-4 was less than for steel 35561 on quenching may have resulted from its much higher residual aluminum content. The high silicon content of steel AS-4 has not produced good creep resistance even when tested in the coarse-grained condition.

TABLE I.—CREEP DATA FOR AISI 1015 STEEL TESTED AT 850 F AND 15,000 PSI.

Steel	Specimen Number	Carbon, per cent	Manganese, per cent	Phosphorus, per cent	Sulfur, per cent	Silicon, per cent	Aluminum		Deoxidation Practice	Heat Treatment	Austenite Grain Size ASTM Number	Creep Rate, per cent per hr	Total Deformation at 500 hr, per cent
							lb per ton	Soluble					
AS-2	AS-2-2	0.15	0.47	0.015	0.024	0.39	1.2	0.047	Si-Al Killed	1950 F, 1½ hr, A. C.	Coarse (2–4)	0.0007	0.557
3194	3194-6	0.15	0.47	0.017	0.025	0.16	1.0	0.001	Si-Al Killed	1900 F, 2 hr, A. C.	Coarse (2–4)	0.000035	0.235
AS-2	AS-2-4	0.15	0.47	0.015	0.024	0.39	1.2	0.047	Si-Al Killed	1750 F, 1½ hr, A. C.	Fine (7–9)	0.0044	3.32
3194	3194-19	0.15	0.47	0.017	0.025	0.16	1.0	0.001	Si-Al Killed	1575 F, 5 min., A. C.	Fine (5–7)	0.000126	0.394
AS-4	AS-4-1	0.16	0.45	0.015	0.024	0.58	2.4	0.088	Si-Al Killed	2000 F, 1½ hr, A. C.	Coarse (2–4)	0.0036	2.23
5280	5280-1	0.17	0.48	0.020	0.03	0.16	2.0	...	Si-Al Killed	2000 F, 1 hr, A. C.	Coarse (2–4)	0.00006	0.165
AS-4	AS-4-4	0.16	0.45	0.015	0.024	0.58	2.4	0.088	Si-Al Killed	1750 F, 6 hr, A. C.	Fine (7–9)	0.0045	2.98
5280	5280-9	0.17	0.48	0.020	0.03	0.16	2.0	...	Si-Al Killed	1730 F, 1 hr, A. C.	Fine (7–8)	0.015	2.767
AS-4	AS-4-7	0.16	0.45	0.015	0.024	0.58	2.4	0.088	Si-Al Killed	2000 F, 1½ hr, W. Q. 1 hr at 950 F	Coarse (2–4)	0.00027	0.280
35561	CC-2-1	0.08	0.36	...	0.025	0.006	2.8	0.025	Al Killed Non-Aging	2225 F, 2 hr, W. Q. 1 hr at 950 F	Coarse (2–4)	0.00004	0.198
35561	CC-1	0.08	0.36	...	0.025	0.006	2.8	0.025	Al Killed Non-Aging	2225 F, 2 hr, A. C.	Coarse (2–4)	0.00102	1.638

If the creep results from Specimens AS-2-2 and AS-4-1, both tested in the coarse-grained condition, are compared, it will be noted that the lower creep rate was obtained from Specimen AS-2-2, which had the lower silicon and aluminum contents. In the fine-grained condition, these steels had similar creep rates.

Both Glen (6) and Bardgett (7) show that as a rule silicon increases the creep resistance and aluminum decreases the creep resistance of carbon steels. Their results differed in the magnitude of the effects noted for silicon and aluminum. Bardgett's tests indicated that the effect of silicon was greater than reported by Glen. His results indicate that when only enough silicon is added to deoxidize the steel the creep resistance, is reduced, but as more silicon is added, in excess of about 0.08 per cent silicon, the creep resistance is improved. This was true up to 0.24 per cent silicon in the steels he tested. And Glen's tests indicated that the effect of aluminum was markedly greater than shown by Bardgett for the steels he used.

If the steels used in this investigation behaved similarly to Glen's, the high residual aluminum contents may have been responsible for the over-all poor creep properties of the steels tested.

Since it was necessary to keep the expenditures for this work to a minimum, the testing was discontinued as soon as the results indicated that higher than normal silicon contents were not producing good creep properties in these aluminum-treated steels. Therefore, the other steels available for this project were not tested.

It is unfortunate that the special test steels produced for this project had such high residual aluminum contents. The analyses of the steels tested by Smith and Dulis (4) strongly suggest that by some different furnace practice, other than used for the special steels reported on here, steels with equal aluminum additions can be made that will have lower residual aluminum contents. Only then could the possible effect on creep resistance of silicon content up to 0.60 per cent in aluminum-treated steels be evaluated and a determination be made as to whether creep-resistant, fine-grained steels could be produced.

SUMMARY

The four special steels used in this study were prepared by mold additions to four ingots of a regular open-hearth heat of AISI 1015 steel. Steels of higher than normal silicon (0.39 and 0.58 per cent Si) were prepared; the first with aluminum additions of 0.35, 1.2, and 2.4 lb aluminum per ton and the latter with 2.4 lb per ton. The aluminum residuals in these steels were much higher than desired or expected.

These special steels, when tested at 850 F and 15,000 psi, showed considerably lower creep resistance than steels with similar aluminum additions and normal silicon content. Relatively little improvement in creep resistance of these steels was produced by coarsening of the austenite grain size, although as in previous experiments, rapid cooling from the coarsening temperature produced some improvement as compared with the properties for slow-cooled material.

Higher than normal silicon contents did not produce good creep properties in AISI 1015 aluminum-treated steels. High aluminum residuals in these steels may have been responsible for the observed effects.

REFERENCES

(1) H. C. Cross and J. G. Lowther, "Progress Report on Study of Effects of Manufacturing Variables on the Creep Resistance of Steels," *Proceedings*, Am. Soc. Testing Mats., Vol. 38, Part I, p. 149 (1938).

(2) H. C. Cross, and J. G. Lowther, "Second Progress Report on Study of Effects of Variables on the Creep Resistance of Steels," *Proceedings*, Am. Soc. Testing Mats., Vol. 40, p. 125 (1940).

(3) H. C. Cross and W. Simmons, "Study of Effects of Variables on the Creep Resistance of Steels," *Proceedings*, Am. Soc. Testing Mats., Vol. 44, p. 161 (1944).

(4) G. V. Smith and E. J. Dulis, "Effect of Manufacturing Practice on Creep and Creep-Rupture Strength of Low-Carbon Steel," *Proceedings*, Am. Soc. Testing Mats., Vol. 49, p. 584 (1949).

(5) M. J. Day and J. B. Austin, "Heat Etching as a Means of Revealing Austenite Grain Size," *Transactions*, Am. Soc. Metals, Vol. 28, pp. 354–68 (1940).

(6) J. Glen, "Abnormal Creep in Carbon Steels," *Journal*, Iron and Steel Inst., Vol. 155, pp. 501–512 (1947).

(7) W. E. Bardgett, Discussion of Glen's paper (6), *Journal*, Iron and Steel Inst., Vol. 157, pp. 579–584 (1947).

APPENDIX II

STABILITY OF STEELS AS AFFECTED BY TEMPERATURE

(PROJECT NO. 29)

PREPARED BY J. J. KANTER AND E. A. STICHA

Graphitization susceptibility of certain casting steels for welded high-pressure, high-temperature steam piping systems has been the subject of extensive study by a committee of the Manufacturers Standardization Society of the Valve and Fittings Industry. In the first part of the program (now concluded), effect of aluminum deoxidation, chromium additions, and post-welding normalizing treatment on the rate of graphite formation in carbon-molybdenum (WC1) steel were investigated. Procedure for testing the various materials has been presented previously together with results of periodic structural examinations.

Some of these earlier results were given in terms of estimated fraction of "contact" zone carbide transformed to graphite in an attempt to obtain a quantitative measure of structural deterioration. However, further experience with this procedure shows that percentage of graphite derived from metallographic study leads to high results, possibly because of carbon diffusion from adjoining areas or presence of ferrite within the graphite nodules. Hoyt and Hall reported similar discrepancies and data of Wilder and associates offer other examples of this sort. Though quantitative aspects of earlier results may not be accurate, the qualitative relations are not changed. While Hoyt and Hall circumvented this difficulty by "calibrating" the method, it was felt such a procedure would be unnecessary for purposes of this study.

Table I contains a summary of 1025 F exposure graphitization susceptibility test results for carbon-molybdenum (WC1) and $\frac{1}{2}$ per cent chromium-molybdenum (WC3) steels. In compiling this information covering about 40 distinct heats of steel, several of which were heat treated in more than one way, somewhat better than 300 metallographic specimens were examined, often at both high and low magnification, over aging periods which extended to 40,000 hr (about $4\frac{1}{2}$ years) in some cases. The approximate time of initial graphite formation (if any) is shown together with general structural condition at conclusion of aging. The findings reported earlier are substantiated further by results of these more extended tests.

The rôle of aluminum in lowering resistance to graphite formation is well known; this behavior has been quite apparent for some time in the WC1 steels where varying amounts of aluminum were added for deoxidation (Groups 1 and 4). More recently it was reported that aluminum-free steel of this type (Group 5) may graphitize also and the latest examinations disclose some graphite in three of these steels. Since the latter steels yield a normal McQuaid-Ehn carburized structure, this test is not an entirely reliable criterion of graphitization susceptibility.

The beneficial effect of chromium additions for conferring carbide stability has been well established. Study of data for

Groups 2 and 3 shows that considerable stability is realized by use of ½ per cent chromium, even with large aluminum additions for deoxidation, but there remains a slight tendency to develop graphite near the weld heat-affected zone. For long-time, high-temperature service without danger of graphitization, higher chromium percentages are indicated.

Normalizing and drawing after welding improves resistance to graphitization of WC1 steels but does not eliminate carbide decomposition as may be noted by Group 6 results.

None of the test specimens developed chain graphite. The low-temperature end of the weld-heat-affected area, that is, the "contact" zone, was the most susceptible location, although in some instances there was as much graphite in the unaffected base metal.

TABLE I.—SUMMARY OF GRAPHITIZATION SUSCEPTIBILITY TEST DATA FOR WC1 AND WC3 STEELS.

	Mark	"Contact" Zone Microstructure after Exposure at 1025 F
Group 1 WC1 Steel (½% Mo) Deoxidized with 2 lb Al per ton	1B	Graphite first noted at 8000 hr, many nodules at 25000 hr
	2B	Graphite first noted at 2000 hr, many nodules at 31000 hr
	4B	Graphite first noted at 1000 hr, many nodules at 25000 hr
	5B	Graphite first noted at 2000 hr, many nodules at 31000 hr
	7B	Graphite first noted at 1500 hr, many nodules at 30000 hr
	7BI	Graphite first noted at 1500 hr, many nodules at 30000 hr
Group 2 WC1 Steel with Cr (½% Mo, ¼% Cr) Deoxidized with 2 lb Al per ton	6B	Graphite first noted at 5000 hr, few more nodules at 31000 hr
	6BI	No graphite at 6000 hr
	7B′	No graphite at 15000 hr
	7*	No graphite at 15000 hr
	7–	Graphite first noted at 6000 hr, more nodules at 15000 hr
	7X	Graphite first noted at 4500 hr, more nodules at 15000 hr
Group 3 WC3 Steel (½% Mo, ½% Cr) Deoxidized with 2 lb Al per ton	1C	No graphite at 25000 hr
	2C	No graphite at 31000 hr
	4C	No graphite at 25000 hr
	5C	No graphite at 31000 hr
	6C	Graphite first noted at 3000 hr, occasional nodules at 9000 hr
	7C	No graphite at 30000 hr
	6CI	No graphite at 6000 hr
	7CI	Graphite first noted at 10000 hr, few more nodules at 30000 hr
	7C′	No graphite at 15000 hr
	7Y	No graphite at 10000 hr
Group 4 WC1 Steel (½% Mo) Deoxidized with ½ lb Al per ton	1A	Graphite first noted at 20000 hr, few isolated nodules at 25000 hr
	2A	Graphite first noted at 9000 hr, more nodules at 25000 hr
	4A	Graphite first noted at 8000 hr, more nodules at 25000 hr
	5A	Graphite first noted at 9000 hr, few more at 31000 hr
	7A	Graphite first noted at 3000 hr, many nodules at 30000 hr
	7AI	Graphite first noted at 3000 hr, many nodules at 30000 hr
Group 5 WC1 Steel (½% Mo) Deoxidized with Si only	3D	No graphite at 40000 hr
	4D	Graphite first noted at 30000 hr, few nodules at 40000 hr
	5D	Graphite first noted at 15000 hr, more nodules at 40000 hr
	6D$_{1s}$	No graphite at 25000 hr
	6DI	No graphite at 6000 hr
	6DI$_{1s}$	No graphite at 6000 hr
	7DI	Graphite first noted at 30000 hr
Group 6 WC1 Steel (½% Mo) Deoxidized with 2 lb Al per ton. After welding, Norm. 1650 F, Drawn 1200 F	1BH	Graphite first noted at 5000 hr, few more nodules at 25000 hr
	2BH	Graphite first noted at 5000 hr, more nodules at 31000 hr
	4BH	Graphite first noted at 5000 hr, few more nodules at 25000 hr
	5BH	Graphite first noted at 4000 hr, many nodules at 31000 hr
	6BH	Graphite first noted at 4000 hr, more nodules at 31000 hr
	7BH	Graphite first noted at 6000 hr, more nodules at 30000 hr
	7BIH	Graphite first noted at 1500 hr, many more nodules at 30000 hr
Group 7 WC3 Steel (½% Mo, ½% Cr) Deoxidized with ½ lb Al per ton	2E	No graphite at 40000 hr
	4E	No graphite at 40000 hr
	5E	No graphite at 31000 hr
Group 8 WC3 Steel (½% Mo, ½% Cr) Deoxidized with Si only	4G	No graphite at 25000 hr
	5G	No graphite at 40000 hr

The second phase of the casting steel program consisted of similar graphitization susceptibility tests applied to more highly alloyed materials, namely, nickel-chromium-molybdenum (WC4) steel, $1\frac{1}{4}$ per cent chromium-molybdenum (WC6) steel, and 2 per cent chromium-molybdenum (C3A) steel. Exposure was at 1100 F rather than 1025 F in an effort to speed up the graphitization process. Results shown in Table II demonstrate the carbide stability of these steels. Approximately 40 more heats are represented in this tabulation, and, again, the specimens examined during the aging period (40,000 hr maximum time) are in excess of 300. Once again effectiveness of $\frac{1}{2}$ per cent chromium in enhancing graphitization resistance is shown by data for additional heats of WC3 steel. In view of the low susceptibility of this material, the possibility of graphite formation in steels of higher chromium content seems quite remote, even in an accelerated test, and this portion of the casting program is being concluded also.

TABLE II.— SUMMARY OF GRAPHITIZATION SUSCEPTIBILITY TEST DATA FOR WC3, WC4, WC6 AND C3A STEELS.

	"Contact" Zone Microstructure
WC3 Steel (½% Mo, ½% Cr) Deoxidized with 2 lb Al per ton	6 samples at 1025 F for 20,000 hr, no graphite 4 samples at 1100 F for 30,000 hr, no graphite 3 samples at 1100 F for 25,000 hr, no graphite 1 sample at 1100 F for 20,000 hr, no graphite 1 sample at 1100 F for 15,000 hr, no graphite 2 samples at 1100 F for 10,000 hr, one contains a very few graphite nodules
WC4 Steel (½% Mo, ½% Cr, 1% Ni) Deoxidized with 2 lb Al per ton	1 sample at 1025 F for 40,000 hr, no graphite 3 samples at 1100 F for 30,000 hr, no graphite 1 sample at 1100 F for 25,000 hr, no graphite 1 sample at 1100 F for 20,000 hr, no graphite 1 sample at 1100 F for 10,000 hr, no graphite
WC6 Steel (½% Mo, 1¼% Cr) Deoxidized with 2 lb Al per ton	2 samples at 1025 F for 20,000 hr, no graphite 2 samples at 1025 F for 15,000 hr, no graphite 2 samples at 1100 F for 30,000 hr, no graphite 3 samples at 1100 F for 25,000 hr, no graphite 2 samples at 1100 F for 20,000 hr, no graphite 1 sample at 1100 F for 15,000 hr, no graphite 3 samples at 1100 F for 10,000 hr, no graphite
C3A Steel (½% Mo, 2% Cr) Deoxidized with 2 lb Al per ton	5 samples at 1025 F for 40,000 hr, no graphite 1 sample at 1025 F for 15,000 hr, no graphite 3 samples at 1100 F for 30,000 hr, no graphite 1 sample at 1100 F for 25,000 hr, no graphite 2 samples at 1100 F for 20,000 hr, no graphite 1 sample at 1100 F for 10,000 hr, no graphite

It is apparent from examination of these specimens that chromium stabilizes carbide structure in another way in that it reduces tendency for agglomeration during elevated temperature exposure. This may be expected to be a factor in the preservation of creep strength. Though the main purpose of this project has been study of structural stability, certain other characteristics of these casting steels have been determined. End-quench hardenability test results, which have found some application for evaluating weldability, are presented in Figs. 1 and 2. An austenitizing and quenching temperature of 2100 F was employed in this work.

Deoxidation treatment has little effect on response to hardening although, on the average, the WC1 steels deoxidized with silicon give only slightly higher hardness values near the quenched end. The effect of increasing chromium content in producing greater hardenability is brought out by the progressively higher levels of the right-hand ends of curves shown in Figs. 1 and 2. Spread of test results is thought to be related primarily to variations in carbon content.

Tensile and hardness data have been obtained for all steels and, in general, requirements of applicable specifications have been met so no detailed accounting of these results will be made. This statement applied to chemical analyses for the second set of casting steels, also. Some WC1 and WC3 material was aged so that effect of exposure on creep characteristics might be evaluated. However, graphitization in these steels resulted in a demand for more stable materials, so this portion of the program was abandoned.

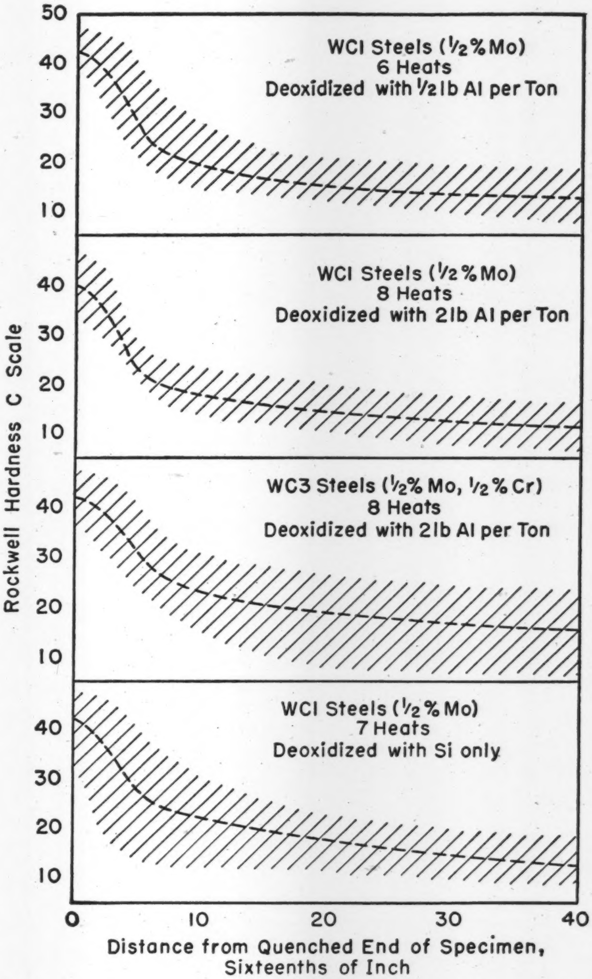

Fig. 1.—End-Quench Hardenability Data for WC1 and WC3 Casting Steels.

Participants[1] in the casting steel study in the way of services or materials include the Chapman Valve Manufacturing Co, Crane Co, Edward Valves, Inc., Lunkenheimer Co, Reading-Pratt & Cady Division of the American Chain & Cable Co, and Walworth Co.

The Pipe Fabrication Institute has investigated the effect of certain preheat, post-heat and welding position variables on the graphitization susceptibility of annealed and normalized $\frac{1}{2}$ per cent chromium-molybdenum (A280) seamless steel pipe. Descriptions of material and procedure have been published previously.[2] To date, after aging at 1100 F for 35,000 hr, during which time about 550 samples were examined, no graphite has been observed in any of the test specimens. This result is in agreement with data for casting steels of similar composition and deoxidation (Group 7). Effect of test variables is, of course, obscured by stability of the carbide in this steel. It was noted that there was less agglomeration of carbides in the annealed samples than in the normalized.

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[1] The authors of this progress report wish to express their appreciation of the hearty cooperation exhibited by these companies which contributed services or materials to the casting steel studies.

The following organizations[3] submitted specimens for this test program: American Locomotive Co, Crane Co, Grinnell Co, M. W. Kellogg Co, and Pittsburgh Piping and Equipment Co.

The investigation of graphitization in steel, being conducted at Battelle Memorial Institute under the sponsorship of Project No. 29 of the Joint ASTM-ASME Committee on Effect of Temperature on the Properties of Metals, has the following objectives: (1) to obtain information on the relationship between the graphitization susceptibility of plain-carbon steel and its aluminum and nitrogen contents, (2) to obtain information on the influence of normal amounts of manganese and silicon on graphitization, and (3) to obtain data on the manner in which chromium increases resistance toward graphitization.

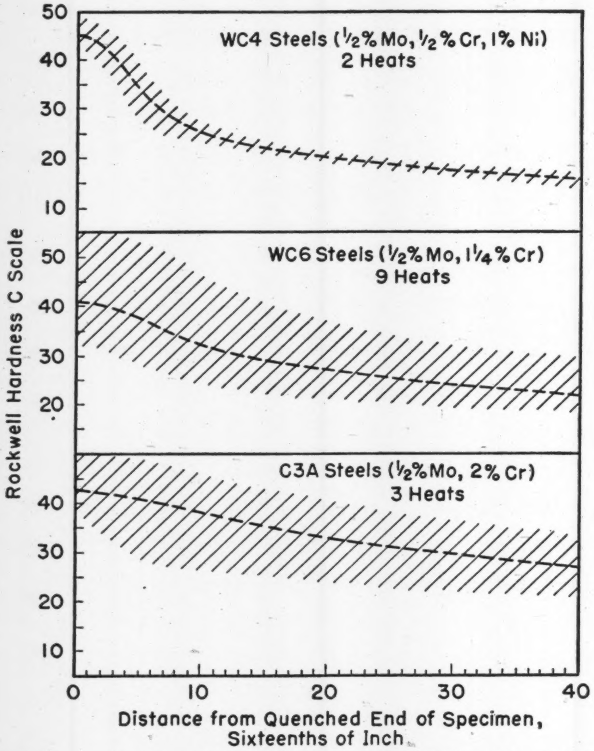

FIG. 2.—End-Quench Hardenability Data for WC4, WC6 and C3A Casting Steels.

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[2] "Stability of Steels As Affected By Temperature (Project No. 29)," *Proceedings*, Vol. 47, p. 207 (1947).

[3] The authors wish to acknowledge the contribution of these organizations, which submitted specimens for this test program, and express their appreciation accordingly.

The initial phases of the preparation of the test material required for the study were completed. Twelve laboratory induction heats of steel, containing controlled amounts of C, N, Al, Mn, Si, Cr, S, and P were melted in an induction melting unit specially constructed for melting under artificial atmospheres. The heats weighed about 2¾ lb each and were about 1½ in. in diameter by 4 in. long. Further work on the project awaits the results of chemical analysis of the heats.

REPORT OF COMMITTEE C-1

ON

CEMENT*

Committee C-1 on Cement held three meetings during the past year: in Atlantic City, N. J., June 28, 1950; in Skokie, Ill., October 10, 1950; and in Cincinnati, Ohio, March 7, 1951.

Through the kind invitation from the Portland Cement Assn., it was possible to hold the October meeting in the Association's new research laboratories at Skokie. The committee members had the opportunity to inspect the laboratories, and the Association arranged for an inspection trip to the site of the long-time exposure tests of concrete at Naperville.

The committee records with sorrow the death of a long-time member, H. D. Baylor.

Committee D-2 on Petroleum Products and Lubricants, and Committee C-1 on Cement are sponsoring jointly the Symposium on Flame Photometry that will be held at the Annual Meeting of the Society this year.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1950 Annual Meeting, Committee C-1 presented to the Society through the Administrative Committee on Standards the following recommendations:

Revision of Tentative Specifications for:

Air-Entraining Portland Cement (C 175 – 48 T)

Revision of Tentative Method of:

Test for Air Content of Air-Entraining Hydraulic-Cement Mortar (C 185 – 49 T).

Tentative Revision of Standard Specifications for:

Portland Cement (C 150 – 49).

These recommendations were accepted by the Standards Committee on September 26, 1950, and the revised tentatives together with the tentative revision of standard C 150 – 49 appear in the 1950 Supplement to Book of ASTM Standards, Part 3.

In addition, Committee C-1 presented to the Society, through the Administrative Committee, a new Tentative Method of Test for Presence of Calcium Sulfate in Hydrated Portland Cement Mortar. The new tentative was accepted on May 11, 1951, and appears in the 1951 Supplement to Book of ASTM Standards, Part 3.

NEW TENTATIVES

Tentative Method of Test for Time of Setting of Hydraulic Cement by the Gillmore Needles.—In line with the recommendation of the revised Vicat test offered later in this report, the committee recommends the acceptance, as a new tentative, of those portions of the Methods of Test for Time of Setting of Hydraulic Cement by the Vicat or Gillmore Needles (C 191 – 49)[1] that relate to the Gillmore test, plus the revisions that are herein set forth. This new tentative[2] is designed to form eventually a separate presentation of the Gillmore test, under a new serial designation, in order to secure increased certainty in the setting time references in the cement specifications. The new tentative is essentially the present Method C 191 modified as follows for the use of the Gillmore needle only:

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* Presented at the Fifty-fourth Annual Meeting of the Society, June 18–22, 1951.

[1] 1949 Book of ASTM Standards, Part 3.

[2] The new tentative was accepted by the Society and appears in the 1951 Supplement to Book of ASTM Standards, Part 3.

General.—Delete all reference to the Vicat test, which will be covered in its revised form by the now proposed revision of Method C 191.

Section 2(c).—Revise "100-ml" to read "150-ml," and delete further reference to 100-ml graduates.

Section 4.—Revise "clean water" to read "mixing water."

Section 6(b).—In the second sentence, replace the portion after the word "indentation" by the words "initial Gillmore needle." In the third sentence, replace the portion after the word "indentation" by the words "final Gillmore needle."

Section 6(c).—Replace its present wording, which is only a reference, by the words to which it refers, the last sentence of Section 5(*c*), which section will be deleted in this proposed revision.

Tentative Definition of Pozzolan.—As the result of considerable deliberation, the committee recommends the acceptance as tentative of the following definition of the term "pozzolan":

> *Pozzolan.*—The term "pozzolan" is used to designate a siliceous or aluminous material, which in itself possesses little or no cementitious value but will, in finely divided form and in the presence of moisture, chemically react with calcium hydroxide at ordinary temperatures to form compounds possessing cementitious properties.

REVISION OF STANDARDS, IMMEDIATE ADOPTION

Standard Specifications for Masonry Cement (**C 91 - 49**).[1]—The committee recommends revision for immediate adoption of this standard, and accordingly asks for a nine-tenths affirmative vote at the Annual Meeting in order that this recommendation may be referred to letter ballot of the Society. It is advantageous to have this revision become effective now in order to provide reference to the Tentative Specifications for Flow Table for Use in Tests of Hydraulic Cement (C 230 - 49 T),[1] and thereby lessen the chance for misunderstanding of the requirements for this equipment in the tests of masonry cement. This change, which would be in line with the last revision in Standard C 109, is as follows:

Section 19.—Replace Paragraphs (*g*) and (*h*) by a new Paragraph (*g*) to read as follows:

> (*g*) *Flow Table.*—The flow table, flow mold and caliper shall conform to the requirements of Tentative Specifications for Flow Table for Use in Tests of Hydraulic Cement (ASTM Designation: C 230).[1]

Reletter subsequent paragraphs accordingly.

REVISIONS OF TENTATIVES

Tentative Specifications for Portland-Blast-Furnace Slag Cement (**C 205 - 48 T**).[1]—The committee recommends that this tentative be revised as outlined below. The first two items are intended to bring the specifications in line with similar recent action in the case of the Tentative Specifications for Air-Entraining Portland Cement (C 175 - 50 T)[3] in the matter of additions.

Section 3(c).—Replace the present sentence with the following sentence: "When air-entraining cement (Type IS-A) is specified, an addition meeting the requirements of Tentative Specifications for Air-Entraining Additions for Use in the Manufacture of Air-Entraining Portland Cement (C 226 - 50 T) shall be used." Delete the current footnote 5 which referred to the sentence that is now being replaced.

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[3] 1950 Supplement to Book of ASTM Standards, Part 3.

Section 7.—Immediately after the third sentence, add the following new sentence: "The manufacturer, upon request, shall furnish the purchaser the identity of the air-entraining addition used."

Table II on Physical Requirements.—Delete footnote *b*. Such a note does not appear in the specifications C 150 and C 175 - 50 T. Delete the tensile strength requirements for Type IS-A cement, thereby presenting the requirements for this cement in closer conformity with the Tentative Specifications for Air-Entraining Portland Cement (C 175 - 50 T).[3]

Tentative Method of Test for Setting Time of Hydraulic Cement in Mortar (**C 229 - 49 T**).[1]—The committee recommends the acceptance of the following revisions in Tentative C 229 - 49 T.

Section 2(e).—Revise the last sentence to read, "The mold for containing the mortar shall be a smooth cylindrical ring 76 ± 0.5 mm (3-in. tubing) in inside diameter by 40 mm in depth."

Section 2(f).—In the last sentence, change dimensions of ring to conform to the above revision in Section 2(*e*).

Section 4(c).—Revise the second sentence to read as follows: "This mortar shall be puddled 14 times with the tamper (Section 2(*d*)) by holding the 1-in. side of the tamper parallel to the radius of the ring and making a complete circle with uniform spacing around the outside of the mold. Then an additional 4 strokes are applied to the center of the specimen with the longitudinal axis of the tamper at the center of the mold and the tamper rotated through 45 degrees between each stroke." Insert the word "then" at the beginning of third sentence. In the fourth sentence revise "30-degree angle" to read "20-degree angle."

Section 4(d).—Add the following at the end of this paragraph: "Trial batches shall be made with varying percentages of water until the desired consistency is obtained. Each trial shall be made with a fresh batch of mortar."

Section 4(e).—Break the fourth sentence into two sentences and change to read as follows: "The penetration shall be checked at every 30 min interval after the mold is filled until the 2-mm needle fails to penetrate to the bottom of the mortar. Then the penetrations shall be checked every 10 min for Type III cement and every 15 min for the other types of cement until a penetration of 10 mm or less is obtained."

TENTATIVE REVISIONS OF STANDARDS

Methods of Test for Time of Setting of Hydraulic Cement (**C 191 - 49**).[1]—In keeping with the explanation, offered earlier in this report, concerning the recommended new tentative Gillmore time-of-setting test under a new serial designation, the committee recommends the acceptance of the proposed Vicat setting-time test, as appended hereto,[4] as a tentative revision of Method C 191. This proposed revision is designed to effect eventually a separate standard presentation of the revised Vicat setting time test.

Standard Method of Test for Fineness of Portland Cement by the Turbidimeter (**C 115 - 42**).[1]—The committee recommends the acceptance of the following tentative revision of this method to permit the use of the one-piece molded glass tank and also the use of aged linseed oil as a dispersing agent. The revision is as follows:

Section 3(d).—Replace the entire section by the following:

> 3 (*d*) *Sedimentation Tank.*—The sedimentation tank shall be either: (*1*) constructed of $\frac{3}{16}$-in. plate glass cemented together to form a

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[4] The tentative revision in the form of a new tentative was accepted by the Society and appears in the 1951 Supplement to ASTM Book of Standards, Part 3.

rectangular tank or (*2*) a molded glass tank having walls approximately $\frac{3}{16}$ in. thick having plane, polished faces. The inside dimensions of the rectangular tank shall be 2 by $1\frac{1}{2}$ in. by 8 in. in height. The permissible variations on the inside dimensions of the tank shall be ±0.03 in. in length and width and ±0.1 in. in height. The 2-in. faces of the tank shall be placed normal to the beam of light, and shall be equidistant within 0.01 in. at all points. A mark shall be placed on the side of the tank to indicate a volumetric content of 335 ml, which is the level to which the tank will be filled in a test. A tank filled to the mark with clear kerosene and placed in the turbidimeter light beam shall have the same microammeter reading within ±0.1 microamperes for the entire usable portion of the tank.

Section 13(*a*).—Replace the present first three sentences by the following: "The cement sample shall be placed in a test tube with 10 to 15 ml of kerosene and 5 drops of the dispersing agent, oleic acid or aged linseed oil (Note 1)." Add the following new Note 1 and renumber the current Note 1, and the reference thereto in the last line of Section 13(*a*), as Note 2.

NOTE 1.—The oleic acid if used shall be of USP grade. The aged linseed oil if used shall have a specific gravity in the range of 0.948 to 0.953. These dispersing agents have slight or no effect on the viscosity of the kerosene. Both dispersing agents change with age and exposure to heat and light and should be kept in well stoppered dark-glass bottles. Where conditions are such that poor dispersion is obtained with oleic acid or when foaming of the suspension occurs (possibly due to traces of water in the kerosene) aged linseed oil shall be used as the dispersing agent. The same dispersing agent shall be used for both the instrument calibration and the fineness determination.

Standard Methods of Chemical Analysis of Portland Cement (**C 114 - 47**).[1]—The committee recommends accepting as tentative revisions[5] the following changes which are essentially those that are now being considered in connection with the revision of Federal methods for analyzing portland cement:

Section 8(*d*).—Revise the first sentence to read as follows: "Treat the SiO_2 thus obtained, which will contain small amounts of impurities, in the crucible with 1 to 2 ml of water, 2 drops of 1:1 H_2SO_4, and about 10 ml of HF, and evaporate cautiously to dryness."

Section 9(*a*).—Between the fourth and fifth sentences insert the following: "In the event difficulty from bumping is experienced while boiling the ammoniacal solution, a digestion period of 10 min on a steam bath or a hot plate, having the approximate temperature of a steam bath, may be substituted for the 50 to 60 sec-boiling period."

At the end of Paragraph (*a*), after the phrase "with hot NH_4Cl (20 g per liter)" add "(Note 4)," referring to the following:

NOTE 4.—Two drops of methyl red indicator should be added to the NH_4Cl solution in the wash bottle followed by 1:1NH_4OH dropwise until the color just changes to yellow. If the color reverts to red at any time due to heating, it should be brought back to yellow by addition of a drop of 1:1 NH_4OH.

Section 13(*a*).—After the seventh sentence which ends "boil until all the bromine is expelled," substitute the following for the remainder of Section 13, except for Paragraphs (*d*) and (*e*):

Add 5 ml of HCl, dilute to 200 ml, add a few drops of methyl red indicator, and 30 ml of warm ammonium oxalate solution (50 g per liter). Heat the solution to 70–80 C, and add NH_4OH (1:1) dropwise with stirring until the color changes from red to yellow. Allow the solution to stand without further heating for one hr (not longer), with occasional stirring during the first 30 min. Filter and wash moderately with cold 0.1 per cent ammonium oxalate solution. Reserve the filtrate and washings.

(*b*) Place the beaker in which the precipitation was made under the funnel, pierce the apex of the filter paper with a stirring rod, place the rod in the beaker, and wash the precipitate into the beaker with a jet of hot water. Dissolve the oxalate remaining in the pores of the paper by thorough washing with 50 ml of hot, dilute HCl (1:4), followed by hot water. Dilute to 200 ml with water, add 20 ml of ammonium oxalate solution, heat the solution nearly to boiling, and precipitate calcium oxalate again by neutralizing the acid solution with ammonium hydroxide as previously described. Allow the solution to stand for an hour or two (standing for 2 hr at this point does no harm), filter and wash as before. Combine the filtrate with that already obtained and reserve for the determination of MgO (Section 15).

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[5] These tentative revisions were amplified and resubmitted to the Administrative Committee on Standards on September 12, 1951, see Editorial Note, p. 243.

(*c*) Dry the precipitate in a weighed, covered platinum crucible, char the paper without inflaming, burn the carbon at as low a temperature as possible, and finally, heat with the crucible tightly covered in an electric furnace or over a blast lamp at a temperature of 1100 to 1200 C. Cool in a desiccator and weigh as CaO. Repeat the ignition to a constant weight."

Delete Note 1 and Note 2.

Section 28(a).—After the first phrase "To 1 g of the sample," add "(Note 1)" referring to the following:

NOTE 1.—If sulfur trioxide is to be determined by turbidimetry, Section 44, then it is permissible to determine the insoluble residue on a half-gram sample. In this event the percentage of insoluble residue should be calculated to the nearest 0.01 by multiplying the weight of residue obtained by 200. However, the cement shall not be rejected for failure to meet the insoluble residue requirement unless a 1-g sample has been used.

The present note of Section 28(*a*) should accordingly be numbered as "Note 2."

"*Section 35(a)*.—Substitute for the present Section 35(*a*) the following:

35. (*a*) NOTE.—See Note 1 of this section and Note 2 of Section 33.

Acidify the combined filtrates obtained in the determination of the ammonium hydroxide group (Section 9) and, if necessary, evaporate to a volume of about 200 ml. Add 5 ml of HCl, a few drops of methyl red indicator, and 30 ml of warm ammonium oxalate solution (50 g per liter). Heat the solution to 70–80 C and add NH_4OH (1:1) dropwise with stirring until the color changes from red to yellow. Allow the solution to stand without further heating for one hr (not longer), with occasional stirring during the first 30 min. Filter and wash the precipitate eight to ten times with hot water. The total amount of water used in rinsing the beaker and washing the precipitate should not exceed 75 ml. Reserve the filtrate for the determination of MgO (Sections 15, 36, or 40). Place the beaker in which the precipitation was made under the funnel, pierce the apex of the filter paper with a stirring rod, place the rod in the beaker, and wash the precipitate into the beaker with a jet of hot water. Drop about ten drops of 1:1 H_2SO_4 around the top edge of the filter paper. Wash the paper five more times with hot water. Dilute to 200 ml, and add 10 ml of 1:1 H_2SO_4. Heat the solution just short of boiling, and titrate with the standard $KMnO_4$ solution to a permanent pink color. Add the filter paper and macerate it. Continue the titration slowly until the pink color persists for 10 sec.

Section 40(b).—In place of the present second sentence substitute the following:

Heat this solution to boiling and boil for 50 to 60 sec. In the event difficulty from bumping is experienced while boiling the ammoniacal solution, a digestion period of 10 min on a steam bath or a hot plate, having the approximate temperature of a steam bath, may be substituted for the 50 to 60-sec boiling period.

In the present third sentence insert, "steam bath" after the word "burner."

In the eighth sentence place a period after the word "precipitation." Then substitute for the remainder of the present paragraph, the following:

Acidify the filtrate and washings, and adjus. the volume to about 200 ml. Add 5 ml of HCl, a few drops of methyl red indicator, and 30 ml of warm ammonium oxalate solution (50 g per liter). Heat the solution to 70–80 C and add NH_4OH (1:1) dropwise with stirring until the color changes from red to yellow. Allow the solution to stand without further heating for one hr (not longer), with occasional stirring during the first 30 min.

Change the first word of Section 40(*c*) from "Cool" to "Heat."

ADOPTION OF TENTATIVE AS STANDARD

Tentative Method of Test for Fineness of Portland Cement by Air Permeability Ap-

paratus (**C 204 - 46 T**).[2]—This tentative has stood for two years, without revision, and the committee now recommends that the tentative, with the following revisions, be adopted as standard. The revisions are as follows:

Section 2(e) on Filter Paper.—In the second sentence just after the word "circular," add the words "with smooth edges."

Section 3(d) on Weight of Sample.—Change the porosity from "0.505 ± 0.005" to read "0.500 ± 0.005" in both places where it appears. This last change is recommended in order to promote reproducibility of results and greater uniformity of test beds, difficulty having been encountered under the present requirements when testing some portland cements with fineness close to the specification limits.

TENTATIVES CONTINUED WITHOUT REVISION

Based on recent and current study in the various related subcommittees, the committee recommends that the following tentatives be retained as tentative without revision at this time:

Tentative Methods of Chemical Analysis of Portland Cement (C 114 - 48 T),
Tentative Method of Test for Sodium Oxide and Potassium Oxide by Flame Photometry (C 228 - 49 T),
Tentative Term Addition as Applied to Hydraulic Cement (C 219 - 48 T),
Tentative Specifications for Flow Table for Use in Tests of Hydraulic Cement (C 230 - 49 T), and
Tentative Specifications for Natural Cement (C 10 - 49 T).

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[6]

ACTIVITIES OF SUBCOMMITTEES

The various subcommittees have been busily engaged in studying their problems, some of which are here briefly recounted.

The Sponsoring Committee on Portland Cement (C. H. Scholer, chairman) is studying suggestions relative to requirements for setting time and fineness. Consideration is being given to certain recommended revisions in the limitations for sulfur trioxide so as to provide for the use of the optimum sulfur trioxide content in a given cement. The adequacy of current strength requirements is being studied.

The Sponsoring Committee on Masonry Cement (H. L. Vanderwerp, chairman) has been studying specifications for masonry and natural cements and has recommended certain revisions in the apparatus requirements in the Standard Specifications for Masonry Cement (C 91 - 49).

The Sponsoring Committee on Blended Cement (L. R. Forbrich, chairman) has been studying the questions of specifications for fly ash, portland - fly ash cement, and blended cement containing calcined reactive siliceous materials.

The Editorial Subcommittee (W. J. McCoy, chairman) has been engaged in reviewing the various C-1 standards, particularly newly proposed texts, and has offered numerous recommendations for their editing.

The Working Committee on Methods of Chemical Analysis (W. C. Hanna, chairman) has continued its studies of methods of interest to the cement chemists and has recommended certain revisions in the current standard methods. The subcommittee, through Leonard R. Pritchard, has aided in the preparations for the flame photometer symposium that will be held during the Society's 1951 Annual Meeting.

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[6] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

The Working Committee on Volume Change and Soundness of Portland Cement (H. F. Gonnerman, chairman) has studied proposals relating to apparatus required in the tests within its scope. A study has been in progress for some time on the autoclave test data for a large number of cements, and a summary of the study will be reported to Committee C-1. The subcommittee is bringing up to date the 1947 summary of various methods of test for reactive aggregates, such revision to be distributed to the members of Committees C-9 and C-1. In cooperation with Subcommittee II-b of Committee C-9 on Concrete and Concrete Aggregates, a bibliography on literature relating to chemical reactions in concrete has been prepared. This bibliography will be sent to all members of Committees C-9 and C-1.

The Working Committee on Sulfate Resistance (D. Wolochow, chairman) has studied the results of tests on lean mortar bars made by 17 cooperating laboratories. The results of a recent "repeat" series of cooperative tests are awaited before the subcommittee submits recommendations.

The Working Committee on Time of Setting (E. E. Berger, chairman) continued its study of methods for determining the time of setting of cement, particularly the methods employing cement pastes. A proposed revision of the standard Vicat test was recommended, and that revision is now being presented to the Society by Committee C-1.

The Working Committee on Heat of Hydration (W. J. McCoy, chairman) sponsored a cooperative test program that is well under way and in which a number of laboratories are investigating certain proposed modifications of the standard test for heat of hydration of portland cement.

The Working Committee on Bleeding, Plasticity and Workability (H. L. Kennedy, chairman) has been considering possible changes in the Tentative Method of Test for Bleeding of Cement Pastes and Mortars, and has been studying possible means of measuring the plasticity of cement pastes and mortars.

The Working Committee on Fineness (R. L. Blaine, chairman) has studied some of the details of the current two methods for determining the fineness of portland cement and has recommended certain revisions therein. Committee C-1 is now offering those recommendations to the Society.

The Working Committee on Strength (G. L. Lindsay, chairman) has been studying questions relating to the equipment used in compression tests of mortar cubes. The subcommittee has also made a study of strength test data from a number of laboratories, and is now engaged in examining the results of recent cooperative test programs in order to secure more information on which to submit to C-1 a method for flexural strength tests of mortars.

The Working Committee on Additions (H. W. Russell, chairman) has been studying the numerous questions that have arisen in connection with that subcommittee's work. The subcommittee has collaborated with Subcommittee III-h of Committee C-9 in the efforts to secure as much uniformity as possible in the specifications for additions and for admixtures, C 226 – 50 T and C 260 – 50 T respectively.

The Working Committee on Coordination of Methods of Test (J. R. Dwyer, chairman) has been engaged in the study of current problems relating to standardization of methods and apparatus. Information of interest to the various subcommittees has been made available to them through this subcommittee.

The Working Committee on SO_3

Content (H. S. Meissner, chairman) continued its study of questions relating to the proposed Tentative Method of Test for Presence of Calcium Sulfate in Hydrated Portland Cement Mortar. The proposed method is appended to this report.[1] The subcommittee has requested the producer members of Committee C-1 to study their cements to determine their optimum gypsum content, making tests at 18 as well as at 24 hr. The subcommittee will distribute a suggested program for such study. An interesting report of the subcommittee "The Optimum Gypsum Content of Portland Cement," was published in the ASTM BULLETIN.[7]

The Working Committee on Methods of Test for Air-Entrainment (H. K. Cook, chairman) has been busily engaged for some time in arranging the details of a very extensive series of cooperative tests in which 11 laboratories are now studying important questions that are involved in measuring the air-entraining properties of cements.

The Subcommittee on Cement Reference Laboratory (G. E. Warren, chairman) reported that the Reference Laboratory had been engaged in the usual inspection of cement testing laboratories throughout the country. Efforts were being made to secure additional personnel. Study was being made of the questions that have arisen in the first general trials of the experimental flow table calibration sample. The Reference Laboratory has aided in editing cement standards and the Manual on Cement Testing, and has handled the usual correspondence relating to inspections and methods of test. The head of the laboratory attended the meetings of Committee C-1 and maintained contact with certain of the ASTM subcommittees that are engaged in the study of questions closely related to the apparatus and methods used in the tests of cement.

This report has been submitted to letter ballot of the committee, which consists of 85 voting members; 73 members have returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

R. R. LITEHISER,
Chairman.

W. S. WEAVER,
Secretary.

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[7] ASTM BULLETIN, No. 169, October, 1950, p. 39.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee C-1 presented to the Society through the Administrative Committee on Standards the following recommendations:

Revision of Tentative Methods of:
- Chemical Analysis of Portland Cement (C 114 – 48 T), and
- Test for Potential Alkali Reactivity of Cement-Aggregate Combinations (C 227 – 50 T).

Revision of Tentative Specifications for:
- Air-Entraining Additions for Use in the Manufacture of Air-Entraining Portland Cement (C 226 – 50 T), and
- Air-Entraining Portland Cement (C 175 – 50 T).

Revision of Tentative Definition of:
- Term Addition as Applied to Hydraulic Cements (C 219 – 48 T).

Tentative Revision of Standard Methods of:
- Chemical Analysis of Portland Cement (C 114 – 47), and
- Test for Autoclave Expansion of Portland Cement (C 151 - 49).

Tentative Revision of Standard Specifications for:
- Portland Cement (C 150 – 49).

These recommendations were accepted by the Standards Committee on September 12, 1951, and the revised tentatives and tentative revisions of the standards appear in the 1951 Supplement to Book of ASTM Standards, Part 3.

On January 16, 1951, the Administrative Committee on Standards accepted the following recommendations submitted by Committee C-1:

Revision of Tentative Specifications for:
Air-Entraining Portland Cement (C 175 - 51 T).

Tentative Revision of Standard Specifications for:
Portland Cement (C 150 - 49).

The revised tentative specifications and tentative revision of C 150 - 49 appear in the 1951 Supplement to Book of ASTM Standards, Part 3.

REPORT OF COMMITTEE C-2

ON

MAGNESIUM OXYCHLORIDE AND MAGNESIUM OXYSULFATE CEMENTS*

Committee C-2 on Magnesium Oxychloride and Magnesium Oxysulfate Cements met two times during the year: on June 29, 1950, in Atlantic City, N. J. and on April 27, 1951, in Washington, D. C.

The committee has lost the services of G. J. Fink as secretary, and, in the interim, W. J. Riley has been appointed as secretary *pro tem.*

Three proposed specifications covering magnesium oxide, magnesium sulfate and magnesium chloride have been recommended by the committee for submission to the Administrative Committee on Standards for publication as tentative.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1950 Annual Meeting, Committee C-2 presented to the Society through the Administrative Committee on Standards, the following tentatives:

Tentative Method of:

Chemical Analysis of Magnesium Sulfate, Technical Grade (C 244 - 50 T),
Chemical Analysis of Magnesium Chloride (C 245 - 50 T),
Physical Testing of Magnesia for Magnesium Oxychloride Cements (C 246 - 50 T),
Test for Ignition Loss and Active Calcium Oxide in Magnesium Oxide for Use in Magnesium Oxychloride Cements (C 247 - 50 T),
Test for Bulk Density of Magnesium Oxychloride Cements (C 248 - 50 T),
Slump Test for Field Consistency of Magnesium Oxychloride Cements (C 249 - 50 T),
Mixing Magnesium Oxychloride Cement Compositions with Gauging Solution (for Preparation of Specimens for Laboratory tests) (C 251 - 50 T),
Test for Linear Contraction of Magnesium Oxychloride Cements (C 252 - 50 T),
Test for Linear Change of Magnesium Oxychloride Cements (C 253 - 50 T),
Test for Setting Time of Magnesium Oxychloride Cements (C 254 - 50 T),
Test for Consistency of Magnesium Oxychloride Cements by the Flow Table (C 255 - 50 T),
Test for Flexural Strength of Magnesium Oxychloride Cements (Using Simple Bar with Two-Point or Single-Point Loading) (C 256 - 50 T), and
Test for Compressive Strength of Magnesium Oxychloride Cements (C 257 - 50 T).

Tentative Specifications and Methods for:

Field Determination of Specific Gravity of Gauging Solutions for Magnesium Oxychloride Cements (C 250 - 50 T).

These recommendations were accepted by the Standards Committee on September 26, 1950, and the new tentatives appear in the 1950 Supplement to Book of ASTM Standards, Part 3.

TENTATIVES ADOPTED AS STANDARD

Committee C-2 recommends that the following three methods be adopted as standard by the Society without revision:

Tentative Methods of:

Sampling Magnesium Oxychloride Compositions and Ingredients (D 237 - 49 T),
Test for Sieve Analysis of Magnesium Oxychloride Compositions, Aggregates and Fillers (C 238 - 49 T), and
Test for Sieve Analysis of Plastic Calcined Magnesia (C 239 - 49 T).

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* Presented at the Fifty-fourth Annual Meeting of the Society, June 18-22, 1951.

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[1]

This report has been submitted to letter ballot vote of the committee, which consists of 20 members; 15 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

L. S. WELLS,
Chairman.

W. J. RILEY,
Secretary pro tem.

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[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee C-2 presented to the Society through the Administrative Committee on Standards the following recommendations:

Tentative Specifications for:
- Oxychloride Magnesia (C 275 - 51 T),
- Magnesium Chloride (C 276 - 51 T), and
- Magnesium Sulfate Epsom Salts, Technical Grade (C 277 - 51 T).

These recommendations were accepted by the Standards Committee on September 12, 1951, and the specifications appear in the 1951 Supplement to Book of ASTM Standards, Part 3.

REPORT OF COMMITTEE C-3

ON

CHEMICAL-RESISTANT MORTARS*

Committee C-3 on Chemical-Resistant Mortars held a meeting at ASTM Headquarters on October 18, 1950. Several subcommittees held meetings preliminary to the main committee gathering. A second meeting of the committee and the subcommittees was held in Cincinnati, Ohio, on March 6, 1951.

At the present time there are 20 members on the committee, of whom 9 are classified as producers, 5 as consumers, 6 as general interest members, and 4 as consulting members.

During the year, the by-laws of the committee were formulated and accepted. To conform with the by-laws, an Executive Subcommittee was elected, composed of the following members: F. O. Anderegg, C. R. Payne, Beaumont Thomas, S. W. Shepard, W. C. Hansen, and V. A. Curll.

NEW TENTATIVE

Committee C-3 recommends that the proposed Tentative Method of Test for Chemical Resistance of Hydraulic Cement Mortars, as appended hereto,[1] be accepted for publication as tentative.

The recommendation appearing in this report has been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

ACTIVITIES OF SUBCOMMITTEES

Considerable progress has been made in the formulation of test methods, specifications and definitions. In addition to the completion of the proposed Tentative Methods of Test for Chemical Resistance of Hydraulic Cement Mortars, methods of test for sulfur mortars are expected to be completed shortly for submission for acceptance as tentative. During the coming year it is anticipated that other test methods will be completed for a major portion of the materials under consideration.

During the year, original experimental work has been undertaken by some of the subcommittees which is reported herein:

Subcommittee III on Resin Mortars (H. B. Staley, chairman) reports extensive work on test methods for resin-base mortars.

Subcommittee VII on Working and Setting Time (R. B. Seymour, chairman) has performed valuable work on the application of various types of apparatus for the determination of working and setting time and the correlation of this information with trowelling methods.

Subcommittee VIII on Bond Strength (J. R. Allen, chairman) has developed a new method for determination of bond strength.

This report has been submitted to letter ballot of the committee, which consists of 23 members; 20 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

F. O. ANDEREGG,
Chairman.

BEAUMONT THOMAS,
Secretary.

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* Presented at the Fifty-fourth Annual Meeting of the Society, June 18–22, 1951.

[1] The new tentative was accepted by the Society and appears in the 1951 Supplement to Book of ASTM Standards, Part 3.

[2] The letter ballot vote on this recommendation was favorable; the results of the vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE C-4

ON

CLAY PIPE*

Committee C-4 held one meeting during the year on June 28, 1950, at the time of the Annual Meeting of the Society.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

On September 26, 1950, the Administrative Committee on Standards accepted the following recommendations submitted by Committee C-4:

Revision of Tentative Specifications for:

Standard Strength Clay Sewer Pipe (C 13 - 44 T), and
Extra Strength Clay Pipe (C 200 - 44 T).

The Specifications for Ceramic Glazed Pipe were accepted as tentative by the Standards Committee on January 17, 1951, and have been assigned the designation C 261 - 50 T.

The new and revised tentatives appear in the 1950 Supplement to Book of ASTM Standards, Part 3.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II on Specifications (J. O. Everhart, chairman).—A task group, appointed to revise the Recommended Practice for Laying Sewer Pipe (C 12 - 19), has completed its draft of a proposed new tentative to be designated as "Recommended Practice for Laying Clay Sewer Pipe," which will replace the old standard.

Subcommittee III on Clay Flue Lining (Wendell Anderson, chairman) has as its major project the drafting of a standard for clay flue linings. Extensive investigations of all the service factors necessary toward writing a standard have been undertaken by the subcommittee.

This report has been submitted to letter ballot of the committee, which consists of 32 members; 25 members returned their ballots, of whom 21 have voted affirmatively and 2 negatively.

Respectfully submitted on behalf of the committee,

J. C. RIEDEL,
Chairman.

R. G. SCOTT,
Secretary.

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* Presented at the Fifty-fourth Annual Meeting of the Society, June 18-22, 1951.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee C-4 presented to the Society through the Administrative Committee on Standards the following recommendations:

Tentative Specifications for:
Extra Strength Ceramic Glazed Clay Pipe (C 278 - 51 T).

Revision and Reversion to Tentative of:
Recommended Practice for Laying Sewer Pipe (C 12 - 19).

These recommendations were accepted by the Standards Committee on September 12, 1951, and the specifications and revised recommended practice appear in the 1951 Supplement to Book of ASTM Standards, Part 3.

REPORT OF COMMITTEE C-7

ON

LIME*

Committee C-7 on Lime held two meetings during the past year: the first at Atlantic City, N. J., on June 30, 1950, and the second at Cincinnati, Ohio, on March 7, 1951.

Members of Committee C-7 have performed a considerable amount of round-robin testing to discover more efficient and accurate methods for determining iron content and settling rates of lime. In addition, Subcommittee IX on Research has been concentrating its activities on the development of a new slaking test on lime. Special task groups are reviewing the Standard Specification for Sand for Use in Plaster (C 35 – 39) and the proposed new tentative mortar specification being recommended by ASTM Committee C-12.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1950 Annual Meeting, Committee C-7 presented to the Society through the Administrative Committee on Standards the following recommendations:

Tentative Specifications for:

Quicklime for Calcium Carbide Manufacture (C 258 – 50 T), and

Hydrated Lime for Grease Manufacture (C 259 – 50 T).

These recommendations were accepted by the Standards Committee on September 26, 1950, and the new tentative specifications appear in the 1950 Supplement to Book of ASTM Standards, Part 3.

On March 2, 1951, the Standards Committee accepted tentative revisions of the Standard Specifications for Hydraulic Hydrated Lime for Structural Purposes (C 141 – 42).

This report has been submitted to letter ballot vote of the committee, which consists of 45 members; 34 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

WALTER C. VOSS,
Chairman.

ROBERT S. BOYNTON,
Secretary.

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* Presented at the Fifty-fourth Annual Meeting of the Society, June 18–22, 1951.

REPORT OF COMMITTEE C-8

ON

REFRACTORIES*

Committee C-8 on Refractories has held two meetings during the past year: the seventy-eighth in State College, Pa., on September 14, 1950, and the seventy-ninth in Cincinnati, Ohio, on March 7, 1951.

The committee lost by death one member, Cecil E. Bates of the Ironton Fire Brick Co.

The present membership of the committee is 39 members; of whom 19 are classified as producers, 12 as consumers, and 8 as general interest members. There are 16 consulting members.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the Annual Meeting, Committee C-8 presented to the Society through the Administrative Committee on Standards the following recommendations:

Tentative Revision of Standard Method for:
Basic Procedure in Panel Spalling Test for Refractory Brick (C 38 – 49),
Panel Spalling Test for High Heat Duty Fireclay Brick (C 107 – 47),
Panel Spalling Test for Super Duty Fireclay Brick (C 122 – 47), and
Spalling Test for Fireclay Plastic Refractories (C 180 – 49).

These recommendations were accepted by the Standards Committee on January 19, 1951, and the tentative revisions appear in the 1950 Supplement to Book of ASTM Standards, Part 3.

NEW TENTATIVES

Committee C-8 recommends the publication as tentative of the following two methods of test, as appended hereto:[1]

Tentative Method of Test for Modulus of Rupture of Castable Refractories, and
Tentative Method of Test for Permanent Linear Change on Firing of Castable Refractories.

These two methods have appeared in the Tentative Specifications for Castable Refractories for Boiler Furnaces and Incinerators (C 213 – 50 T).[2] Their publication as tentative represents a separation of the test methods from the specifications for greater flexibility of use.

TENTATIVE REVISIONS OF STANDARDS

Standard Definitions of Terms Relating to Refractories (**C 71 – 49**).[2]—The committee recommends the following tenattive revision of this standard:

Delete the definition under Spalling of Refractories, including the note, and replace it with the following four definitions:

Spalling of Refractories.—The cracking or rupturing of a refractory unit, which usually results in the detachment of a portion of the unit.

Mechanical Spalling of Refractories.—The spalling of a refractory unit caused by stresses resulting from impact or pressure.

Structural Spalling of Refractories.—The spalling of a refractory unit caused by stresses resulting from differential changes in the structure of the unit.

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* Presented at the Fifty-fourth Annual Meeting of the Society, June 18–22, 1951.

[1] The new tentatives were accepted by the Society and appear in the 1951 Supplement to Book of ASTM Standards, Part 3.

[2] 1949 Book of ASTM Standards, Part 3.

[3] 1950 Supplement to 1949 Book of ASTM Standards, Part 3.

Thermal Spalling of Refractories.– The spalling of a refractory unit caused by stresses resulting from non-uniform dimensional changes of the unit produced by a difference in temperature.

ADOPTION OF TENTATIVE AS STANDARD

Tentative Specifications for Castable Refractories for Boiler Furnaces and Incinerators (**C 213 – 50 T**).[2]—Having received no criticism or proposal bearing on the essential features of these specifications, the committee recommends for reference to letter ballot of the Society for adoption as standard this tentative, subject to editorial separation of the test methods in Sections 9(*b*), 9(*c*), and 9(*d*) from the main body of the standard which shall consist of Sections 1 to 9(*a*), inclusive.

REVISION OF STANDARD, IMMEDIATE ADOPTION

Committee C–8 recommends for immediate adoption the following revision and accordingly asks for a nine-tenths affirmative vote at the Annual Meeting in order that these revisions may be referred to letter ballot of the Society:

Standard Methods of Chemical Analysis of Refractory Materials (**C 18 – 45**).[2]—

Section 1(*a*).—In the first sentence revise "and magnesite" to read "magnesite, and dolomite."

Section 1(*b*).—Change "Magnesite Refractories:" to "Magnesite and Dolomite Refractories:".

Section 3.—Revise the last sentence to read "Fine grinding shall be done in a suitable mortar (agate, mullite, alumina, or boron carbide) to prevent the introduction of impurities."

Section 4.—Revise the first sentence to read "Except in the case of dolomite refractories (NOTE), moisture shall be determined on the sample in its ordinary air-dried condition."

Add the following note:

NOTE.—An exception is made in the case of dolomite refractories (see Sections 42 and 44), on which the determination of free moisture is omitted, and on which percentage compositions are reported on the ignited basis.

Section 40.—Change the heading preceding Section 40 to read "Magnesite and Dolomite Refractories."

Section 41.—Renumber as 41(*a*) and introduce the paragraph with the words "For magnesite refractories, a 10-lb sample . . .".

In the third sentence revise "an agate mortar" to read "a suitable mortar (agate, mullite, alumina, or boron carbide)" and after the word "container," insert "(e. g. a tightly rubber-stoppered bottle)."

Add the following paragraph as 41(*b*):

(*b*) For dolomite refractories, two samples are required. These shall consist of the following: (*1*) A sample for the determination of the loss on ignition shall consist of 10 lb carefully obtained and then reduced as rapidly as possible to pass completely a No. 16 (1190-micron) sieve by one pass through a suitable mill. This crushing must be done in 3 min or less. The crushed material shall be split repeatedly without delay (preferably in a mechanical splitter) until a 4-oz sample results. This shall be placed immediately in an air-tight container (e.g. a tightly rubber-stoppered bottle). (*2*) A sample for the determination of the SiO_2, Fe_2O_3, Al_2O_3, CaO, and MgO shall be prepared as described in Section 41 (*a*)."

Section 42.—Revise this section to read:

42. For magnesite refractories, weigh 1.00 g of the sample and heat to constant weight at a temperature not under 105 nor over 110 C. Record the loss in weight as moisture.

NOTE.—The determination of moisture in dolomite refractories is omitted, since no free moisture exists in these products. Further, they are sometimes coated with sealing oil or tar.

Section 43.—Revise this section to read as follows:

43. (*a*) For magnesite refractories, weigh 1.00 g of the moisture-free (105 to 110 C) sample and heat to constant weight over a blast lamp, or in an electric furnace, at 900 to 1000 C. Record the loss in weight as the ignition loss.

(*b*) For dolomite refractories, weigh approximately 10 g of the specially prepared sample (Section 41 (*b*)) quickly into a tared porcelain crucible and heat to constant weight over a blast lamp, or in an electric muffle furnace, at 900 to 1000 C. The loss in weight is calculated to percentage loss on ignition.

New Sections 44 and 45.—Add new Sections 44 and 45 to read as follows:

44. *Statement of Analysis for Dolomite Refractories.*—Prepared samples of dolomite refractories are known to pick up moisture and CO_2 on storage in the laboratory. For this reason the determinations which follow are reported on the ignited basis. The loss on ignition value to be used shall be determined on the material passing the No. 100 (149-micron) sieve (Section 41 (*b*) (*2*)). This determination shall be made on the same day the portions for chemical analysis are weighed.

45. *Character of the Sample to be Weighed.*—In the following sections, the sample weighed shall be as follows:

(*a*) *Magnesite.*—Use a portion of the material passing the No. 100 (149-micron) sieve (Section 41 (*a*)) which has been dried to constant weight at 105 to 110 C.

(*b*) *Dolomite.*—Use a portion of the material passing the No. 100 (149-micron) sieve (Section 41 (*b*) (*2*)).

Section 44.—Renumber as Section 46. In the first sentence delete the words "moisture-free (105 to 110 C)." Change the fourth sentence to read as follows: "Cool, uncover, **Caution** (see Note), add 8 ml of perchloric acid, $HClO_4$ (60 to 70 per cent), and then evaporate to heavy white fumes of $HClO_4$."

Add the following note:

NOTE: **Caution.**—Boiling perchloric acid is violently explosive when contaminated by easily oxidizable material or organic matter. When analyzing dolomite refractories containing sealing oil or tar, 5 to 10 ml of HNO_3 (sp gr 1.42) are added just *before* the $HClO_4$ to oxidize the organic materials, and eliminate the danger of a perchloric acid explosion.

Section 45.—Renumber as Section 47.

Section 46.—Renumber as Section 48. In the first sentence, delete "moisture-free (105 to 110 C)."

Section 47.—Renumber as Section 49. Change Note to read as follows:

NOTE.—In the complete analysis of magnesite refractories, these may include P_2O_5, TiO_2 and small amounts of SiO_2, present in such small amounts as to be disregarded. Dolomite refractories sometimes contain appreciable amounts of TiO_2, in which case it should be determined, and a correction made for the Fe_2O_3 and Al_2O_3 determined according to Sections 48 and 49.

Section 48.—Renumber as Section 50. In the first sentence of Paragraph (*b*), delete the words "moisture-free (105 to 110 C)." At the beginning of Footnote 8 insert the words "Applicable to magnesite refractories only."

Section 49.—Renumber as Section 51.

Section 50.—Renumber as Section 52. In the first sentence delete the words "moisture-free (105 to 110 C)."

Sections 51 to 62, inclusive.—Renumber consecutively as Sections 53 to 64.

ADOPTION OF TENTATIVE REVISIONS AS STANDARD

Standard Definitions of Terms Relating to Refractories (**C 71 - 49**).[3]—The committee recommends adoption as standard of the tentative revisions covering new definitions for Carbon Refractory and Carbon-Ceramic Refractory.

Standard Specifications for Refractories for Malleable Iron Furnaces with Removable Bungs, and for Annealing Ovens (**C 63 - 47**).[3]—The committee recommends adoption as standard of the tentative revision pertaining to Section 11(*i*).

Standard Specifications for Refractories for Heavy Duty Stationary Boiler Service (**C 64 - 48**).[2]—The committee recommends adoption as standard of the tentative revision pertaining to Section 6(*g*).

Standard Specifications for Refractories for Incinerators (**C 106 - 48**).[2]—The committee recommends adoption as standard of the tentative revision pertaining to Section 5(*c*).

Standard Specifications for Refractories for Moderate Duty Stationary Boiler Service (**C 153 – 48**).[2]—The committee recommends adoption as standard of the tentative revision pertaining to Section 5(*b*).

The recommendations in this report have been submitted to letter ballot of the committee the results of which will be reported at the Annual Meeting.[4]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Industrial Survey (R. P. Stevens, chairman) reports on four new or revised surveys on the following subjects: Incinerators, a new survey prepared by R. H. Stellwagen with assistance from Kay Hesselberg; Continuous Plate Glass and Window Glass Furnaces, revised by A. H. Couch; Lead Industry, revised by A. P. Thompson and R. E. Illidge; and Malleable Iron Air Furnace, revised by R. A. Witschey. Nine other surveys are on the agenda.

Subcommittee II on Research (R. E. Birch, chairman) has presented a report, by D. R. Torgeson, on third-point loading as contrasted with the customary center-point loading. The subject will be considered further by Section III (*A*) on Load.

Subcommittee III on Tests (S. M. Phelps, chairman) has approved the recommendations concerning methods of test given earlier in this report.

Section A on Load (L. J. Trostel, chairman) has reviewed recent work on sonic testing with a view to its possible application to refractories.

Section B on Spalling (R. S. Bradley, chairman) finds no significant difference in results when 2-in. brick are used in a panel in place of 2½ or 3-in. brick.

Section C on Temperature (J. L. Carruthers, chairman) has made a preliminary report on the results of comparative Pyrometric Cone Equivalent tests in 28 laboratories, with 6 standard samples prepared by the Refractories Fellowship at Mellon Institute.

Section F on Tests for Refractory Insulation (W. L. Stafford, chairman), with the aid of 3 laboratories, is examining the effect of rate of load application on the measured strength of refractory insulators and insulating refractories.

Section G on Porosity and Permanent Volume Change (S. M. Swain, chairman) has begun a comparative study of methods now in use for measuring bulk density of calcined materials.

Subcommittee IV on Heat Transfer (C. L. Norton, Jr., chairman) has compared thermal conductivity measurements on Group 20 Insulating Fire Brick in 2 laboratories, with the standard apparatus, and found results in satisfactory agreement, with deviations of not over 3 to 5 per cent.

The Editorial Subcommittee VIII (R. B. Sosman, chairman) has made a number of minor, though significant, changes in wording, which need not be reported here in full, as they will be incorporated in the new Manual. The most important of these changes is the separation of Standard C 213 into three parts: a specification standard C 213 consisting of the present Sections 1 to 9 (*a*), and two test methods, recommended earlier in this report for acceptance as tentatives.[1] A new edition of the Manual of ASTM Standards on Refractory Materials is in preparation; it will be in the hands of the Society as soon as the Industrial Surveys have been satisfactorily revised. With the committee's approval, the new Manual is planned to include new information on standard samples of refractories and on standard clay minerals, and a glossary originally assembled for the American Refractories Institute.

Subcommittee XI on Special Refrac-

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[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

tories (H. Wilson, chairman) is beginning an undertaking of a kind heretofore untried by the committee, namely, the solicitation of funds from the refractories industry for a 3- to 5-yr program of research on the testing of products not now subject to standard tests, such as the mullite group of bonded refractories, electric furnace cast refractories, zircon refractories, and basic granular refractories. The tests proposed for study include reheat changes, high-temperature load, spalling, and slag attack.

A special subcommittee (J. D. Sullivan, chairman) has looked into current practice in ASTM with respect to definitions, classifications, and specifications, and has found considerable divergence among committees. A foundation has been laid for better delineation of the scope of these three types of standards in Committee C-8.

This report has been submitted to letter ballot of the committee, which consists of 39 members; 39 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

R. B. SOSMAN,
Chairman.
R. S. BRADLEY,
Vice-Chairman.

W. R. KERR,
Secretary.

REPORT OF COMMITTEE C-9

ON

CONCRETE AND CONCRETE AGGREGATES*

Committee C-9 on Concrete and Concrete Aggregates held three meetings last year: on June 27, 1950, during the Annual Meeting of the Society in Atlantic City, N. J.; on October 10, 1950, at the new Research Laboratory of the Portland Cement Assn in Skokie, Ill.; and on March 7 in Cincinnati, Ohio, during the Spring Committee Week of the Society. While the committee has held these three well-attended meetings and has been active, there is relatively little to report requiring action by the Society; however, many recommendations covering new standards and modifications in present standards are in prospect.

On May 31, 1951, the committee suffered the loss through death of an honorary member, R. W. Crum. He had been a member since 1923 and served as chairman from 1932 to 1938.

The committee takes great pleasure in announcing the election of F. E. Richart and M. O. Withey as honorary members. Professor Richart has participated actively in the work of the committee since 1922 and served as chairman for the term 1944–1946. Dean Withey was a charter member of the committee and has been active in its work since that time.

In 1938 the committee established the Sanford E. Thompson Award in honor of its first chairman, who continued active in the work of the committee until his death in February, 1949. That award is made for a paper of outstanding merit on concrete and concrete aggregates presented before the Society. This year the Sanford E. Thompson Award will be made to R. C. Mielenz, L. P. Witte, and O. J. Glantz of the U. S. Bureau of Reclamation for their paper on "Effect of Calcination on Natural Pozzolans."[1]

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1950 Annual Meeting, Committee C-9 presented to the Society through the Administrative Committee on Standards the following recommendations:

Tentative Specifications for:
Air-Entraining Admixtures for Concrete (C 260 – 50 T).
Revision of Tentative Methods of:
Testing Air-Entraining Admixtures for Concrete (C 233 – 49 T).

These recommendations were accepted by the Standards Committee on September 26, 1950, and the new and revised tentatives appear in the 1950 Supplement to Book of ASTM Standards, Part 3.

REVISION OF TENTATIVE

Tentative Method of Test for Fundamental Transverse Frequency of Concrete Specimens for Calculating Modulus of Elasticity (Sonic Method) **(C 215 – 47 T)**[2]:

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* Presented at the Fifth-fourth Annual Meeting of the Society, June 18–22, 1951.

[1] *Symposium on Use of Pozzolonic Materials in Mortars and Concretes*, Am. Soc. Testing Mats., p. 43 to 92, (1949). (Symposium issued as separate publication *STP* No. 99).

[2] 1949 Book of ASTM Standards, Part 3.

The committee recommends that this test method be revised as follows:

Title.—Insert "Young's" before "Modulus of Elasticity".

Section 1.—In the first sentence revise the word "beams" to read "prisms" and insert "Young's" before "Modulus of Elasticity".

Section 2 (a).—Change the third sentence to read as follows:

> The combined oscillator and amplifier shall be capable of delivering at least 5 watts power output with not more than 5 per cent distortion and shall be provided with a means of controlling the output.

Section 2 (b).—Revise to read:

> "Pickup Circuit.—The pickup circuit shall consist of a pickup unit, an amplifier, and an indicator. The pickup unit shall generate a voltage proportional to the amplitude, velocity, or acceleration of the test specimen, and the vibrating parts shall be small in mass as compared to the mass of the test specimen. Either a piezoelectric or magnetic pickup unit meeting these requirements may be used. The amplifier shall have a controllable output of sufficient magnitude to actuate the indicator. The indicator shall consist of a voltmeter, milliammeter, or cathode-ray oscilloscope. For routine testing of specimens whose fundamental frequency may be anticipated within reasonable limits a meter-type indicator is satisfactory and may be more convenient to use than a cathode-ray oscilloscope. It is, however, strongly recommended that whenever feasible a cathode-ray oscilloscope be provided for supplementary use or to replace the meter-type indicator. The use of an oscilloscope as an indicator may be necessary when specimens are to be tested for which the fundamental frequency range is unpredictable. The oscilloscope is also valuable for checking the equipment for distortion and drift and for use in the event that it should be desired to use the equipment for certain other purposes than those specifically contemplated by this method of test. The response of the pickup unit shall be proportional to the motion of the test specimen in accordance with the characteristics of the type of pickup selected, and shall be free from spurious resonances in the normal operating range."

Section 3 (b).—Revise Section 3 (*b*) by changing the tolerances from "plus or minus 1 per cent" and "plus or minus 2 per cent" to "$\pm\frac{1}{2}$ per cent" and "± 1 per cent" respectively.

Section 4 (b).—Revise to read:

> The test specimen shall be forced to vibrate at varying frequencies. At the same time, the indication of the amplified output of the pickup shall be observed. The frequency of the test specimen that results in a maximum indication having a well-defined peak on the indicator (Note) shall be recorded as the fundamental transverse frequency. The amplifiers in the driving and pickup circuits shall be adjusted to provide a satisfactory indication. To avoid distortion, the driving force shall be maintained as low as is feasible for good response at resonance.
>
> NOTE.—Vibrations of the test specimens will be a maximum at the ends, approximately, three-fifths of the maximum at the center, and zero at the nodal points; therefore, movement of the pickup along the length of the specimen will inform the operator whether the vibrations observed in the indicator are from the specimen vibrating in its fundamental transverse mode.

Figure 1.—Revise Fig. 1 as shown:

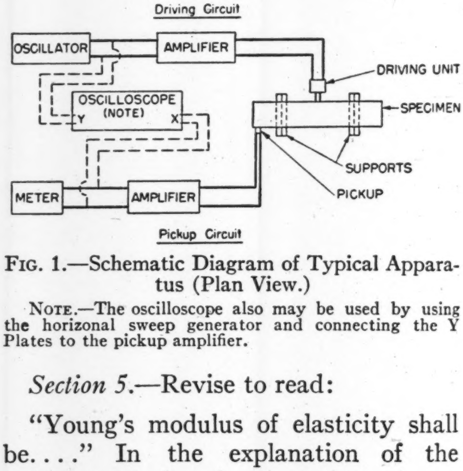

FIG. 1.—Schematic Diagram of Typical Apparatus (Plan View.)

NOTE.—The oscilloscope also may be used by using the horizonal sweep generator and connecting the Y Plates to the pickup amplifier.

Section 5.—Revise to read:

"Young's modulus of elasticity shall be. . . ." In the explanation of the terms used in the equations insert "Young's" before "modulus of elasticity."

Table I.—Add the following to Footnote *a*:

> Poisson's ratio for water saturated concrete may be higher than 1/6. The correction factor,

T', for a different value of Poisson's ratio, μ, and a given K/L, may be calculated from the following relationship:

$$T' = T\left[\frac{1 + (0.26\mu + 3.22\mu^2)K/L}{1 + 0.1328K/L}\right]$$

Where T is taken from Table I for the given K/L.

ADOPTION OF TENTATIVE REVISION AS STANDARD

Standard Method of Test for Abrasion of Coarse Aggregates by Use of the Los Angeles Machine (**C 131 – 47**).[2]—The committee recommends that the tentative revision of this standard, issued in 1949, be referred to letter ballot of the Society for adoption as standard.

CONTINUATION OF TENTATIVES WITHOUT REVISION

The committee recommends that the following eight tentatives, which have stood without revision for two or more years, be retained as tentative without revision:

Tentative Specifications for:
Waterproof Paper for Curing Concrete (C 171 – 49 T).
Tentative Methods of Test for:
Soundness of Aggregates by Use of Sodium Sulfate or Magnesium Sulfate (C 88 – 46 T),
Water Retention Efficiency of Methods for Curing Concrete (C 156 – 44 T),
Waterproof Paper for Curing Concrete (C 171 – 49 T),
Air Content (Volumetric) of Freshly Mixed Concrete (C 173 – 42 T),
Air Content of Freshly Mixed Concrete by the Pressure Method (C 231 – 49 T),
Bleeding of Concrete (C 232 – 49 T),
Comparing Concretes on the Basis of the Bond Developed with the Reinforcing Steel (C 234 – 49 T), and
Soft Particles in Coarse Aggregates (C 235 – 49 T).

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

SUBCOMMITTEE ACTIVITIES

The work of Committee C-9 is carried out by three groups of subcommittees: Group I, Administration (Fred Hubbard, chairman); Group II, Research (A. T. Goldbeck, chairman); and Group III, Specifications and Test Methods (R. R. Litehiser, chairman).

The Subcommittee on Papers and Symposia has been responsible for a large proportion of the program of the 1951 Annual Meeting devoted to concrete and concrete aggregates.

The Subcommittee on Editorial and Definitions has been active in reviewing and clarifying standards and will have some recommendations with respect to definitions to offer during the forthcoming year.

Group II on Research has cooperated with the standards-writing group (Group III) in several projects. The Subcommittee on Chemical Reaction of Aggregates in Concrete (II-b) has presented for consideration various test methods for measuring the effects of chemical reactions between aggregate and cement. The Subcommittee on Dynamic Testing of Concrete (II-e) has also cooperated; in 1947 it prepared a Method of Test for Fundamental Transverse Frequency of Concrete Specimens for Calculating Modulus of Elasticity (Sonic Method) (C 215 – 47 T). Since then further consideration has been given to the test method and revisions have been formulated which are referred to elsewhere in this report.

The Subcommittee on Aggregate Mineralogical Characteristics as Related to Concrete (II-f) has developed two standards which are expected to be recommended for adoption in the near future. One of these is a "Description of the Constituents of Natural Mineral Aggregates" which was published as information in the ASTM BULLETIN.[4] The other is a method for petrographic examination of mineral aggregates, to be based on a paper entitled, "Method of Petrographic Examination of Aggregates for Concrete,"[5] presented by Katharine and Bryant Mather at the 1950 Annual Meeting of the Society.

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[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

In Group III on Specifications and Test Methods, the Subcommittee on Methods of Testing Concrete for Strength (III-a) has had presented to it information indicating that compression tests of concrete specimens made in paper molds may not always be reliable. Accordingly it has in hand the preparation of a tentative specification covering paper molds. The Subcommittee on Methods of Testing Fresh Concrete (III-c) has in preparation a revision of the current Method of Sampling Fresh Concrete and a consolidation of the various methods for measuring the air content of freshly mixed concrete.

The Subcommittee on Specifications for Concrete Aggregates (III-d) has completed a comprehensive revision of the Standard Specifications for Concrete Aggregates (C 33) which is expected to be recommended as a replacement for the current standard during the year. The proposed revision differs from the current standard principally in that specific limitations are recommended throughout instead of requiring the user to state limitations to meet local conditions. The Subcommittee on Lightweight Aggregates (III-f) has arrived at a draft of a proposed specification on which, it is hoped, action can be taken during the coming year.

The Subcommittee dealing with Admixtures (III-h) has reviewed existing standards and has proposed modifications in several existing test methods and the development of some new test methods for use in evaluating admixtures. The Subcommittee on Testing Concrete for Bleeding (III-j) has continued its study of the current test method (C 232 - 49 T); further tests of the method are under way and it is expected that the tentative method will be advanced to standard in the near future.

Three new subcommittees have been created during the year: III-m on Methods of Testing Concrete for Resistance to Abrasion; III-n on Methods of Testing Setting Time of Concrete; and III-o on Methods of Testing Concrete for Resistance to Freezing and Thawing. While these Subcommittees have been organized and their work is under way, it has not advanced sufficiently to permit of comment at this time.

This report has been submitted to letter ballot of the committee, which consists of 87 voting members plus 7 honorary members having voting privileges; 68 members returned their ballots, of whom 63 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

K. B. WOODS,
Chairman.

STANTON WALKER,
Secretary.

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[4] ASTM Bulletin, No. 171, January, 1951, p. 32.

[5] *Proceedings*, Am. Soc. Testing Mats., Vol. 50, p. 1288 (1950).

REPORT OF COMMITTEE C-12

ON

MORTARS FOR UNIT MASONRY*

Committee C-12 on Mortars for Unit Masonry held two meetings during the past year: on June 29, 1950, in Atlantic City, N. J., and on March 9, 1951, in Cincinnati, Ohio.

At the present time, the committee consists of 36 members, of whom 14 are classified as producers, 10 as consumers, and 12 as general interest members. There are 4 consulting members.

The officers for the two-year term 1950–1952 are as follows:

Chairman, J. M. Hardesty.
1st Vice-Chairman, F. O. Anderegg.
2nd Vice-Chairman, P. M. Woodworth.
Secretary, Harry C. Plummer.

NEW TENTATIVE

The committee recommends the publication as tentative of the proposed Tentative Specifications for Mortar for Unit Masonry, appended hereto.[1]

CONTINUATION OF TENTATIVE WITHOUT REVISION

The committee recommends that the Tentative Specifications for Mortar for Reinforced Brick Masonry (C 161 - 44 T) be retained as tentative without revision. The reason for this recommendation is that, when the proposed Tentative Specifications for Mortar for Unit Masonry have stood for one year, it will be desirable either to withdraw the present Specifications C 161 or to revise them to conform to the requirements of the general mortar specifications.

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the annual meeting.[2]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee III on Specifications and Methods of Test for Mortar (R. E. Copeland, chairman) prepared the proposed Tentative Specifications for Mortar for Unit Masonry. These specifications are a result of discussions by the committee extending over a period of approximately 12 years.

Working Subcommittee on Efflorescence (P. L. Rogers, chairman).—Members of this subcommittee are investigating methods of determining efflorescence tendencies of mortars. Progress reports of these studies have been made to the committee.

This report has been submitted to letter ballot of the committee, which consists of 36 members; 25 returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

J. M. HARDESTY,
Chairman.

HARRY C. PLUMMER,
Secretary.

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* Presented at the Fifty-fourth Annual Meeting of the Society, June 18–22, 1951.

[1] The new tentative was accepted by the Society and appears in the 1951 Supplement to Book of ASTM Standards, Part 3.

[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE C-13

ON

CONCRETE PIPE*

Committee C-13 on Concrete Pipe held one meeting during the past year in St. Louis, Mo., on December 7 and 8, 1950. During the year some eight subcommittees or task groups have been active on a variety of subjects pertaining to suggested revisions in the four ASTM standard specifications on concrete pipe.

The present membership of the committee consists of 30 members; of whom 15 are classified as producers, 12 as consumers, and 3 as general interest members.

TENTATIVE REVISIONS OF STANDARDS

Committee C-13 recommends that the following tentative revisions of standards be accepted for publication by the Society:

Standard Specifications for Concrete Sewer Pipe (**C 14 - 41**).[1]—The committee recommends the publication of the following tentative revision:

Section 3.—Change the words "hydrostatic tests" to read "permeability test."

Section 5.—Replace the present Section 5 on "Cement" by the following:

> 5. Portland Cement shall conform to the requirements of the Standard Specifications for Portland Cement (ASTM Designation: C 150);[1] or shall be Air-Entraining Portland Cement conforming to the requirements of the Tentative Specifications for Air-Entraining Portland Cement (ASTM Designation: C 175);[2] or shall be Portland Blast-Furnace Slag Cement conforming to the requirements of the Tentative Specifications for Portland Blast-Furnace Slag Cement (ASTM Designation: C 205).[1]

Section 7.—Revise the first sentence by adding the words "so sized and" after the words "The aggregates shall be." Retain the remainder of the sentence unchanged. After the first sentence add "Admixtures or blends may be used with the approval of the consumer."

Table I.—Delete the word "Average" and replace by the word "Minimum" in the heading of the second column of Table I.

Section 9.—Change the heading from "Hydrostatic Test" to read "Permeability Test." Revise the first sentence to read: "When subjected to the permeability test as specified in Section 19, the pipe shall show no leakage."

Section 10 (b).—Revise the first sentence by adding the words "crushing and absorption" after the words "specimens for."

Section 14 (b).—In the first sentence delete the words "to the top bearing block." After the first sentence add the following: "The load shall be applied through a rigid steel member capable of distributing the load equally throughout the entire length of the 6 by 6-in. wooden bearing block." In the second sentence delete "to the upper bearing block."

Figure 1.—Revise Fig. 1 to show an I-beam in both the cross-sectional view and the end view. The 6 by 6-in. block and the I-beam shall extend the full length of the pipe over the bell exclusive of the socket.

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* Presented at the Fifty-fourth Annual Meeting of the Society, June 18-22, 1951.

[1] 1949 Book of ASTM Standards, Part 3.

[2] 1950 Supplement to 1949 Book of ASTM Standards, Part 3.

Section 15.—Letter the present section as Paragraph (*a*). In the third sentence after the words "at least 6 by 6-in. in cross-section" add the phrase "and may be shaped to conform to the bell of the pipe."

In the fifth sentence add "not exceeding one inch in width" after the words "for the inequalities of the pipe barrel."

In the seventh sentence add "and may be shaped to conform to the bell of the pipe" after the words "straight and true from end to end."

Add the following as Paragraph (*b*):

(*b*) If mutually agreed upon by the manufacturer or other seller and the purchaser, proven types of bearing, such as hard rubber blocks or sand-filled high pressure hose, may be used in lieu of wooden bearings as specified.

Section 18.—Change the heading from "Hydrostatic Test" to read "Permeability Test." Delete the first sentence and replace it with the following: "Sound, full-size pipe not exceeding 2 per cent of each size furnished shall be tested for leakage under the permeability test."

Section 19.—Delete the boldface heading "Hydrostatic Test" and replace it with the heading "Permeability Test."

Revise Section 19 (*a*) to read as follows:

19 (*a*).—The test shall be performed by placing the section of pipe with the spigot end down on a soft rubber mat or its equivalent, weighted if necessary, and filled with water to a level of the base of the socket. The period of test shall be 15 min.

Section 20.—Under the heading of the section "Acceptance or Rejection" add a sub-heading and new Paragraph (*a*) as follows:

20 (*a*) *Acceptance or Rejection as to Strength Properties.*—Pipe shall be acceptable under the strength tests when all test specimens conform to the test requirements. Should any of the preliminary test specimens provided for in Section 10 (*b*) fail to meet the test requirements, then the manufacturer will be allowed a retest on two additional specimens for each specimen that failed, and the pipe shall be acceptable only when all these retest specimens meet the strength requirements.

Under a new sub-heading reading "Acceptance or Rejection as to Other Than Strength Properties" add the present Paragraph 20 (*a*) and reletter as Paragraph (*b*). Reletter the present Paragraph (*b*) as (*c*) and change the word "Average" to read "Minimum."

Section 21.—At the end of Section 21, after Table III, add the following paragraph.

Manufacturers may submit to the purchaser for approval, designs other than those in Tables II and III, including designs for tongue and groove pipe. In no alternate tongue and groove design however, shall the shell thickness be less than 1¾ in., nor shall the test requirements be less than prescribed in Table I.

Standard Specifications for Reinforced Concrete Sewer Pipe (**C 75 - 41**).[1]—The committee recommends the publication as a tentative revision of the following changes:

Section 5.—Make the same revision as described for Section 5 of Specifications C 14 - 41.

Section 7.—After the first sentence add the following sentence: "Admixtures or blends may be used with the approval of the consumer."

Section 8.—Add the words "so sized and" after the words "The aggregates shall be," and retain the remainder of the sentence without change.

Section 13.—Revise the second sentence to read as follows: "If welded, the member at either a welded splice or intersection shall develop a tensile strength of not less than 52,500 psi."

Section 23.—After the fourth sentence add the following new sentence:

If requested by the manufacturer or the purchaser prior to the test, before the pipe is placed, a fillet of plaster of Paris and sand thick enough to compensate for the inequalities of the pipe barrel, not exceeding one inch in width, shall be cast on the surface of the lower bearings.

After the fifth sentence which reads, "The upper bearing shall be a rigid wooden block straight and true from end to end," add the following sentence:

> The load shall be applied through a rigid steel member capable of distributing the load equally throughout the entire length of the 6 by 6-in. wooden bearing block.

Revise the sixth sentence to read as follows:

> The upper and lower bearings shall extend the full length of the pipe exclusive of the socket and may be shaped to conform to the bell of the pipe.

At end of section add the following paragraph:

> If mutually agreed by the manufacturer or other seller and the purchaser, proven types of bearing such as hard rubber blocks or sand-filled high pressure hose may be used in lieu of wooden bearings as specified.

Figure 2.—Revise Fig. 2 to show an I-beam resting on the wooden bearing block, and the load applied to the I-beam in both the cross-sectional view and the end view. The 6 by 6-in. block and the I-beam shall extend the full length of the pipe over the bell exclusive of the socket.

Figure 3.—Change Fig. 3 to show an I-beam resting on the wooden bearing block, and the load applied to the I-beam in both the cross-sectional view and the end view.

Section 30 (b).—At the end of the sentence add the phrase "in the barrel of the pipe."

Standard Specifications for Reinforced Concrete Culvert Pipe (**C 76 – 41**).[1]—The committee recommends the publication of the following changes as a tentative revision:

Section 5.—Make the same revision as described for Section 5 of Specifications C 14 – 41.

Section 8.—After the words "The aggregates shall be" in the first sentence, add the words "so sized and." After the first sentence add a new sentence to read: "Admixtures or blends may be used with the approval of the consumer."

Section 13.—Revise the second sentence to read: "If welded, the member at either a welded splice or intersection shall develop a tensile strength of not less than 52,500 psi."

Section 25.—Revise as indicated for revision of Section 23 of Specifications C 75 – 41.

Figures 2 and 3.—Revise as indicated for Figs. 2 and 3 of Specifications C 75 – 41.

Section 32 (b).—At the end of the sentence add the phrase "in the barrel of the pipe."

Standard Specifications for Concrete Irrigation Pipe (**C 118 – 39**).[1]—Revise as follows:

Section 5.—Make the same revision as described for Section 5 of Specifications C 14 – 41.

Section 6.—Delete the second sentence.

Section 7.—After the words "The aggregates shall be" in the first sentence, add the words "so sized and." After the first sentence add a new sentence to read: "Admixtures or blends may be used with the approval of the consumer."

Section 13 (a).—In the second paragraph after the seventh sentence which ends with the phrase "true from end to end," add a new sentence to read: "The load shall be applied through a rigid steel member capable of distributing the load equally throughout the entire length of the 6 by 6-in. wooden bearing block." At the end of Section 13 (*a*), add the following paragraph:

> If mutually agreed upon by the manufacturer or other seller and the purchaser, proven types of bearing, such as hard rubber blocks or sand-filled high-pressure hose, may be used in lieu of wooden bearings as specified.

Figure 1.—Revise to show an I-beam resting on the wooden bearing block, and the load applied to the I-beam in both the cross-sectional view and end view.

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

ACTIVE COMMITTEE PROJECTS

Various projects that have received attention in the committee are as follows:

Review of Steel Requirements.—A special committee has reviewed the desirability of revising requirements for steel reinforcement in Specifications C 75 and C 76 to bring these into conformity with the simplified practice published by the U. S. Department of Commerce. In the light of this review, it has been agreed that no changes should be made in the specifications.

Absorption Test.—A subcommittee has been appointed, consisting of E. F. Kelley, chairman, R. R. Litehiser, and A. V. Bratt, to make a study of the absorption test requirements.

Cooperation with the AASHO.—A subcommittee consisting of E. F. Kelley, chairman, H. F. Peckworth, H. W. Easterly, Jr., and R. R. Litehiser has been appointed to confer with the American Association of State Highway Officials with respect to the following:

1. The desirability of eliminating the ultimate failure test from the concrete pipe specifications.
2. The desirability of eliminating the sand bearing test, and
3. The desirability of using cores for testing the compressive strength of the concrete in concrete pipe.

Recommended Practice on Bedding and Backfilling.—In view of the intimate relationship between the strength of pipe, the load it is called upon to carry, and the method of bedding and backfilling, a subcommittee composed of M. W. Loving, chairman, Samuel A. Greeley, and H. F. Peckworth has been appointed to prepare a draft of a Recommended Practice for Bedding and Backfilling of Concrete Pipe Lines.

Long-Range Planning Committee.—A Long-Range Planning Committee has been active for some time, composed of E. F. Kelley, chairman, W. E. Corbett, Samuel A. Greeley, Max Nearing, and H. F. Peckworth. The following items have been referred to the committee:

1. The use of cores for testing the compressive strength of the concrete in concrete pipe.
2. Correlation of wording as between ASTM Specifications C 118, C 14, C 75, and C 76.
3. Correlation of the steel sizes as between Specifications C 75 and C 76 (one specification states the total amount of steel to be used in two lines of circular reinforcement, whereas the other specifies the amount of steel to be used in each line of circular reinforcement).
4. Correlation of the test loads and the required steel in both Specifications C 75 and C 76.
5. Study and possible revision of the ratio between the 0.01 crack and the ultimate failure in Specification C 76.

Reinforced-Concrete Low-Head Pressure Pipe Specification.—A subcommittee has been formed and delegated the task of preparing a draft of such a specification. The subcommittee is composed of Howard G. Curtis, chairman, E. F. Kelley, Samuel A. Greeley, J. A. Dunn, M. W. Loving, and Henry A. Weigand.

This report has been submitted to letter ballot vote of the committee, which consists of 31 members; 23 members have returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

W. W. HORNER,
Chairman.
E. F. KELLEY,
Vice-Chairman.

HOWARD F. PECKWORTH,
Secretary.

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[3] The letter ballot vote on these recommendations was favorable; the results of this vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE C-15

ON

MANUFACTURED MASONRY UNITS*

Committee C-15 and its subcommittees have been extremely active during the past year. Two meetings were held: one on June 27, 1950, at Atlantic City, N. J., in conjunction with the Annual Meeting of the Society, and one on March 9, 1951, in Cincinnati, Ohio, during the ASTM Committee Week.

At the present time Committee C-15 consists of 70 members, of whom 30 are classified as producers, 20 as consumers, and 20 as general interest members.

M. H. Allen has replaced J. H. Lee as secretary of the committee. Subcommittees IV on Paving Brick and V on Sewer Brick have been combined and are known as Subcommittee IV on Paving and Sewer Brick.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the Annual Meeting, Committee C-15 presented to the Society through the Administrative Committee on Standards, the following recommendations:

Revision of Tentative Specifications for:

Structural Clay Facing Tile (C 212 – 49 T), and
Glazed Masonry Units (C 126 – 44 T).

Tentative Revisions of Standard Specifications for:

Concrete Building Brick (C 55 – 37),
Structural Clay Floor Tile (C 57– 49),
Hollow Load-Bearing Concrete Masonry Units (C 90 – 44),
Structural Clay Load-Bearing Wall Tile (C 34 – 49),
Structural Clay Non-Load-Bearing Tile (C 56 – 49),
Hollow Non-Load-Bearing Concrete Masonry Units (C 129 – 39), and
Solid Load-Bearing Concrete Masonry Units (C 145 – 40).

Tentative Revision of Standard Methods of:

Sampling and Testing Concrete Masonry Units (C 140 – 39).

Revision and Reversion to Tentative of Standard Specifications for:

Drain Tile (C 4 – 24).

These recommendations were accepted by the Standards Committee on September 26, 1950, and the new and revised tentatives together with the tentative revisions of the standards appear in the 1950 Supplement to Book of ASTM Standards, Part 3.

ADOPTION OF TENTATIVE AS STANDARD

Committee C-15 recommends that the Tentative Specifications for Vitrified Clay Filter Block for Trickling Filters (C 159 – 48 T)[1] be approved without change for reference to letter ballot of the Society for adoption as standard.

ADOPTION OF TENTATIVE REVISIONS AS STANDARD

The committee recommends that the tentative revisions, pertaining to the title and Section 3 of Standard Specifications for Sand-Lime Building Brick (C 73 – 39)[1], be approved for adoption as standard.

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* Presented at the Fifty-fourth Annual Meeting of the Society, June 18–22, 1951.

[1] 1949 Book of ASTM Standards, Part 3.

EDITORIAL REVISIONS

Standard Specifications for Clay Building Brick (**C 62 – 50**).[2]—Revise Section 2 (*f*) on Strength to read as follows:

> (*f*) When brick are required having strengths greater than prescribed in Paragraphs (*a*) or (*d*), the purchaser should specify minimum strength according to the classification given in Table II.

Standard Methods of Sampling and Testing Structural Clay Tile (**C 112 – 36**).[1]—Add to the end of the first paragraph in Section 10 (*a*) the following:

> After the cap has sufficiently hardened to permit handling of the unit, this procedure shall be repeated with the opposite dry, shellacked, bearing surface. Care shall be taken that the capped surfaces so formed are approximately parallel.

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

This report has been submitted to letter ballot of the committee, which consists of 70 members; 58 members have returned their ballots, of whom 56 have voted affirmatively and 0 negatively (2 "not voting").

Respectfully submitted on behalf of the committee,

J. W. WHITTEMORE,
Chairman.

M. H. ALLEN,
Secretary.

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[2] 1950 Supplement to Book of ASTM Standards, Part 3.

[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee C-15 presented to the Society through the Administrative Committee on Standards proposed Tentative Specifications for Chemical-Resistant Masonry Units. The specifications were accepted by the Standards Committee on September 12, 1951, and appear in the 1951 Supplement to Book of ASTM Standards, Part 2, bearing the designation C 279 – 51 T.

REPORT OF COMMITTEE C-16

ON

THERMAL INSULATING MATERIALS*

Committee C-16 on Thermal Insulating Materials and its subcommittees held two meetings during the past year. One meeting was held at Asbury Park, N. J., on October 23, 24 and 25, 1950, and the other was held at Cincinnati, Ohio, on March 6, 7 and 8, 1951.

The committee, as of February 12, consisted of 67 voting members, 21 nonvoting members, and 2 consulting members. The voting membership is classified as follows: 31 producers, 21 consumers, and 15 general interest members.

The officers of the committee remain the same except that R. P. Sturgis has resigned as secretary and is succeeded by H. G. Hill; also, C. M. Weinheimer replaces A. B. Fisher as member-at-large on the Executive Subcommittee.

The committee passed on a change in the By-Laws, Article III, Section V, whereby no member is permitted to belong to more than two subcommittees with the exception of administrative subcommittees.

The committee sponsored a Symposium on Thermal Insulating Materials at the Spring Meeting of the Society in Cincinnati, Ohio. Five papers and their discussions composed this presentation which was organized by a special subcommittee with Ray Thomas as chairman. This symposium will be issued as a separate publication.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1950 Annual Meeting, Committee C-16 presented to the Society, through the Administrative Committee on Standards, recommendations for publication of the following:

Tentative Specifications for:[1]

Mineral Wool Felt Insulation (Industrial Type),

Mineral Wool Batt Insulation (Industrial Type), and

Mineral Wool Blanket Insulation (Metal-mesh Covered) (Industrial Type).

These recommendations were accepted by the Standards Committee on May 4, 1951, and will appear in the 1951 Supplement to 1949 Book of ASTM Standards, Part 3.

ADOPTION OF TENTATIVES AS STANDARD

Committee C-16 recommends that the Tentative Method of Test for Flexural Strength of Preformed Block Type Thermal Insulation (C 203 – 45 T)[2] be approved without revision for reference to letter ballot of the Society for adoption as standard.

ADOPTION OF TENTATIVE REVISION AS STANDARD

The committee recommends that the tentative revision[2] of the Standard Definitions of Terms Relating to Thermal Insulating Materials (C 168 – 50)[3] be approved for reference to letter ballot of the Society for adoption as standard.

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* Presented at the Fifty-fourth Annual Meeting of the Society, June 18-22, 1951.

[1] The new tentatives were accepted by the Society and appear in the 1951 Supplement to Book of ASTM Standards, Part 3.

[2] 1949 Book of ASTM Standards, Part 3.

[3] 1950 Supplement to 1949 Book of ASTM Standards, Part 3.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends that the following tentative methods and tentative revision, which have stood for two years or more without revision, be continued as tentative:

Tentative Methods of Test for:

Water Vapor Permeability of Sheet Materials used in Connection with Thermal Insulation (C 214 - 48 T),
Thermal Conductance and Transmittance of Built-Up Sections by means of the Guarded Hot Box (C 236 - 49 T).

Tentative Revision of:

Method of Test for Bulk Density of Thermal Insulating Cement (C 164 - 44), and
Methods of Test for Covering Capacity and Volume Change Upon Drying of Thermal Insulating Cement (C 166 - 45).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

ACTIVITIES OF SUBCOMMITTEES

Most subcommittees report considerable progress. Many of the projects have been under continuous study and investigation for several years in an endeavor to approach efficient conclusions. Some of the accomplishments during the past year consisted of the completion of proposed tentative specifications and methods for acceptance by the committee, in addition to those covered in previous recommendations in this report. These include: Method of Test for Determination of the Density of Preformed Block Type Thermal Insulation, Method of Test for Determination of the Density of Preformed Pipe Covering, Specification for Mineral Wool Pipe Insulation (Molded Type), and Specification for Mineral Wool Pipe Insulation (Blanket Type).

This report has been submitted to letter ballot of the committee, which consists of 64 voting members; 63 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

E. R. QUEER,
Chairman.

H. G. HILL,
Secretary.

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[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee C-16 presented to the Society through the Administrative Committee on Standards the following recommendations:

Tentative Specifications for:

Mineral Wool Pipe Blanket-Type Insulation (C 280 - 51 T), and
Mineral Wool Pipe Molded-Type Insulation (for Elevated Temperatures) (C 281 - 51 T).

These recommendations were accepted by the Standards Committee on December 13, 1951, and the specifications appear in the 1951 Supplement to Book of ASTM Standards, Part 3.

REPORT OF COMMITTEE C-18

ON

NATURAL BUILDING STONES*

Committee C-18 held a meeting in Washington, D. C., on March 23, 1951, which was preceded by meetings of the subcommittees. In addition Subcommittee I, Advisory, met on February 20, 1951, in Washington, D. C.

The membership of Committee C-18 now consists of 23 members, of whom 7 are classified as producers, 9 as consumers, and 7 as general interest members. There are also 2 consulting members.

The committee is particularly pleased to record the conferring of the ASTM Award of Merit on one of its members, D. W. Kessler, Chief, Building Stone Section, National Bureau of Standards.

A report has been received covering observations and measurements on the exposure test wall at approximately two years after erection, which is appended hereto.

All standards under the jurisdiction of the committee have been reviewed and the committee presents recommendations as noted in the following paragraphs.

ADOPTION OF TENTATIVE AS STANDARD

The following tentative is recommended for adoption as standard, with a minor addition which is considered editorial in nature:

Tentative Method of Test for Abrasive Resistance of Stone Subjected to Foot Traffic **(C 241 - 50 T)**[1]

Section 8.—Add the following sentence at end of the section: "The report shall identify the type and grade of stone, its source, and the approximate date of removal from the quarry."

TENTATIVES CONTINUED WITHOUT REVISION

Committee C-18 recommends that the following tentatives be continued in their present status without revision:

Tentative Methods of:

Test for Durability of Slate for Roofing (C 217 - 48 T), and

Test for Combined Effect of Temperature Cycles and Weak Salt Solutions on Natural Building Stone (C 218 - 48 T).

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II on Nomenclature and Definitions (L. W. Currier, chairman).—Further study is to be given to grain size and texture nomenclature and a report made at the next meeting. The secretary will contact the industry, securing from each producer a sample of his stone (with polished and broken-rock face), giving his concept of the texture or grain size of the specimen.

The need and demand for definitions of finish: just exactly what constitutes a polished, a honed, and other types of finish has been suggested and the subcommittee will undertake a study of this subject, if possible, in cooperation with Sectional Committee A91 on Granite of the American Standards Association.

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* Presented at the Fifty-fourth Annual Meeting of the Society, June 18-22, 1951.

[1] 1950 Supplement to ASTM Book of Standards, Part 3.

[2] The letter ballot vote on these recommendations was favorable; the results of this vote are on record at ASTM Headquarters.

Subcommittee III on Test Procedures (D. W. Kessler, chairman).—The subcommittee, by unanimous vote, recommended to continue for another year, in tentative status, the Tentative Method of Test for Durability of Slate for Roofing (C 217 – 48 T) and the Tentative Method of Test for Combined Effect of Temperature Cycles and Weak Salt Solutions on Natural Building Stone (C 218 – 48 T).

Subcommittee III is cooperating with Subcommittee IV in the preparation of specifications for roofing slate; it plans to test samples, to be submitted by the slate producers, during the present year.

A report was received from Mr. Lelong of the Mellon Institute, who is currently working on the subject of test procedures for exterior marble for the National Association of Marble Producers. Committee C-18, through the Chairman of Subcommittee III, has offered to assist this project and to duplicate or parallel tests with Mr. Lelong using the facilities of the National Bureau of Standards.

Subcommittee IV on Specifications (F. S. Eaton, chairman).—The proposed specification for roofing slate, which had previously been submitted to the membership for study, was tabled for a year to secure additional data and enable the slate producers to have more time properly to consider the proposed specification.

The proposed specification for exterior marble, which had been tabled for one year, was tabled for an additional six months in order to enable the National Association of Marble Producers, now actively engaged in an extensive research program, to have more time in which to secure tangible results from their studies, and to permit this national association to correlate and compare their research with that previously conducted by Subcommittee IV of Committee C-18.

Subcommittee V on Research (J. R. Shank, chairman).—A request has been received for a specification for limestone as a building material. The limestone producers will be circularized on this subject and consulted on the need for a specification and their ideas as to what it should be.

A question on the problem of determining whether a cement is non-staining or not has been referred by correspondence to ASTM Committees C-1, C-3, and C-12.

This report has been submitted to letter ballot of the committee, which consists of 23 voting members; 20 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

L. W. CURRIER,
Chairman.

F. S. EATON,
Secretary.

APPENDIX

REPORT ON OBSERVATIONS AND MEASUREMENTS ON THE EXPOSURE TEST WALL AT APPROXIMATELY TWO YEARS AFTER ERECTION

Prepared by D. W. Kessler[1]

Discolorations

Marbles.—The marble samples have not been affected by any of the brown discolorations which have shown up so prominently on some other types. Some marbles of variegated colors have shown the characteristic fading effect. This is believed to be caused by a weathered film on the surface. Rehoning of the surface removes the film and restores the color.

White or gray streaks extending downward from mortar joints cause some disfiguration on occasional samples. This is attributed to hydroxide of lime being dissolved by water and deposited on the surface where it is carbonated to form a thin crust of calcium carbonate. Such crusts also appear on other types of stone, and those on sandstone or granite can be readily removed with acid; effervescence indicates their calcareous nature.

Granites.—A small number of the granite samples supplied by the National Museum have developed brown discolorations which resemble iron rust. None of the larger samples from modern or recent quarries have shown any appreciable discolorations. Thin samples set in the concrete on the back of the wall have shown no tendency to stain. Several of these had iron rust left on the back to determine if it would later penetrate to the exposed face. Such slabs have retained their color as well as control samples from which the rust was cleaned.

Limestones.—Few of the limestones have developed discolorations. Two small blocks supplied by the Museum are showing brown stains and two coping blocks have soiled considerably from surface accumulations. Some overhanging trees at the back of the wall may be the cause of the latter. Generally the limestones have retained their original condition.

Sandstones.—Several sandstones have developed brown stains, and some have, apparently, stained adjacent blocks and the surrounding mortar. Such performance has been confined to the small blocks from the Museum; the larger samples from present operations have not shown any discolorations other than a slight amount of efflorescence in isolated portions. Since this type is not known to be appreciably affected by discolorations in structures, it seems likely that those samples which developed pronounced stains may have been stone from near the surface of the ground where organic matter from decaying vegetation was absorbed in considerable amounts.

Since one of the main items under consideration in this test was to compare discolorations obtained when stones are set in lime mortar and cement mortar, a count has been made of discolorations in the two types of mortar. This showed a few more discolorations on the cement mortar portion of the wall. However, a casual examination gives the impression that there are more discolorations on the lime portion. This is probably due to the fact that the lime mortar half of the wall is considerably lighter in color as a whole and, hence, the stains stand out in contrast. The darker appearance of the cement mortar portion is, apparently, due to the fact that it could not dry out as readily after setting or after rains as could the lime mortar section. The reason for this is believed to be that the cement mortar is more impermeable than the lime mortar. One large area near the center of the cement mortar masonry has remained darker than the surrounding part from the beginning. It gives the appearance of being continually damp. This suggests that a permeable mortar for setting stonework has an advantage so far as discolorations are concerned. It also indicates that where an impermeable mortar is used one might overcome some of the discolorations by leaving weep-holes in the mortar at a sufficient number of places to drain off excess accumulations of water that may gain access back of the stonework.

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[1] Chief, Building Stone Section, National Bureau of Standards.

Observations on Mortars

Several cracks have been noted in the lime mortar and few have been found in the cement mortar. All of the coping joints filled with lime mortar have cracked, while 3 out of 8 coping joints filled with cement mortar have cracked. The lime mortar is hard only at the surface. This outer portion can easily be broken through with the point of a knife and the rest raked out like sand.

Weathering of Stone Samples

Twelve samples of stone have been noted that are scaling off in thin layers or eroding along weak veins. The samples include 5 sandstones and 7 limestones. The nature of the disintegration indicates that it is caused by crystallization of salts in the pores. Most of the samples affected are dense and strong materials which would not be expected to suffer from frost action during a short exposure. Sandstone blocks in the quoins, and limestones in the coping, representing modern products, have not shown any ill effects from weathering.

Dimensional Changes

Five sets of measurements have been made on five courses including the base and coping. Each course being measured has 3 reference plugs, one near either end and one at the center. The base course is carefully measured with a precision tape and the lengths of other courses are obtained from this length with a transit. The purpose is to determine if a continual increase in dimension occurs.

In making the measurements it was not feasible to take them all at the same temperature. Actually the lowest temperature of measurement was 62 F and the highest was 86 F. This range is sufficient to affect the results and no accurate way of correcting for temperatures is available because the thermal expansion coefficients of many of the materials are not known. By using an estimated value of this coefficient and correcting the measurements of the original and final lengths the following estimate of length changes has been made: The base course has increased in length by 0.004 ft or 0.011 per cent and the coping increased 0.010 ft over that of the base or a total of 0.037 per cent. Intermediate courses have changed so little that definite statements cannot be made as to their movements.

Two sets of warp measurements have been made but the changes were inappreciable.

REPORT OF COMMITTEE C-19

ON

STRUCTURAL SANDWICH CONSTRUCTIONS*

Committee C-19 on Structural Sandwich Constructions held one meeting on October 26–27, 1950, at the U. S. Forest Products Laboratory, Madison, Wis. Since the last report four new members have been elected and two members have resigned. The present committee consists of 59 members, with 48 voting members, of whom 21 are classified as producers, 11 as consumers, and 16 as general interest members.

E. C. Hartmann has found it necessary to resign the secretaryship of the committee, a task he has ably handled since the committee was formed in 1946; R. B. Dunwody was elected as his replacement. The committee and subcommittee organization has not changed otherwise.

The committee has adopted as satisfactory for measuring thermal conductivity of structural sandwich construction core materials, the Standard Method of Test for Thermal Conductivity by Means of the Guarded Hot Plate (C 177 – 45) developed by Committee C-16 on Thermal Insulating Materials.

NEW TENTATIVES

Committee C-19 recommends that the following three methods and definitions be accepted for publication as tentative, as appended hereto:[1]

Tentative Methods of:

Test for Water Absorption on Core Materials for Structural Sandwich Constructions.
Test for Density of Core Materials.
Shear Test of Sandwich Constructions in Flatwise Plane.

Definition of Terms Relating to:

Structural Sandwich Constructions.

The proposed tentatives have been submitted for review to Committee E-1 on Methods of Testing or to Committee E-8 on Nomenclature and Definitions.

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Mechanical Properties of Basic Materials (R. C. Platow, chairman).—During the year tentative methods of test have been prepared for: density, water absorption, dimensional stability, thermal expansion, and bondability. The methods for determination of density and water absorption have been approved by the entire committee and have been recommended for publication as tentative, as noted earlier in this report. The other methods are being reviewed, and although several seem to be generally satisfactory to the committee, further revisions are being made.

Subcommittee II on Mechanical Properties of Basic Sandwich Construction

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* Presented at the Fifty-fourth Annual Meeting of the Society, June 18–22, 1951.

[1] The new tentatives were accepted by the Society and appear in the 1951 Supplement to Book of ASTM Standards, Part 3.

[2] The letter ballot vote on these recommendations was favorable; the results of this vote are on record at ASTM Headquarters.

(E. W. Kuenzi, chairman).—Test methods for shear flatwise and compressive properties have been developed and approved and are being recommended for publication as tentative. Methods of testing for tension and bending flatwise properties are being revised subsequent to letter ballot.

Subcommittee III on Permanence, Durability, and Simulated Service (D. G. Reid, chairman).—Mr. G. M. Rapp has been appointed chairman of a group charged with developing an outdoor exposure test cubicle design.

Subcommittee IV on Nomenclature and Definitions (J. J. Lamb, chairman).—Definitions have been developed as shown in the appended proposals for adoption as tentatives. Ten other definitions are being resubmitted to letter ballot.

This report has been submitted to letter ballot of the committee which consists of 48 voting members; 41 returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

A. G. H. DIETZ,
Chairman.

R. B. DUNWODY,
Secretary.

REPORT OF COMMITTEE C-20

ON

ACOUSTICAL MATERIALS*

Committee C-20 on Acoustical Materials held one meeting during the year at the Massachusetts Institute of Technology, Cambridge, Mass., on November 13, 1950.

The committee now consists of 51 members, of whom 21 are classified as producers, 13 as consumers, and 17 as general interest members.

The committee has no recommendations to the Society at the present time but has gone on record as adopting two existing ASTM standards without change as being suitable for use for measuring thermal properties of acoustical materials. These two methods are the Standard Methods of Test for Conductivity of Materials by Means of the Guarded Hot Plate (C 177 - 45) and the Tentative Method of Test for Thermal Conductance and Transmittance of Built-Up Sections by Means of the Guarded Hot Box (C 236 - 49 T). The committee is sponsoring a Symposium on Acoustical Materials at this Annual Meeting.[1]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Sound Absorption (H. J. Sabine, chairman) is studying the reverberation chamber, box, and impedance tube methods of measurement. Of these, the last named method should be ready for recommendation as tentative within the next year. The other methods require considerably more research and development.

Subcommittee II on Fire Resistance (Wallace Waterfall, chairman) is investigating improvements in the Columbia or Panel Test described in Federal Specification SS-A-118a, with a view to bringing about improvements and preparing a tentative method.

Subcommittee III on Maintenance (Peter Chrzanowski, chairman) has recommended the undertaking of research projects on cleaning, painting and dirt precipitation.

Subcommittee IV on Application (L. F. Yerger, chairman) is studying test methods for acoustical material adhesives, including a proposed Federal Specification.

Subcommittee V on Basic Physical Properties (W. A. Jack, chairman) has recommended the adoption of the Thermal Conductivity Test methods noted above. A proposed test method for light reflection, based on the Baumgartner Sphere, is being circulated for comments. Various strength tests are being considered.

This report has been submitted to letter ballot of the committee which consists of 51 members; 29 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

H. A. LEEDY,
Chairman.

H. J. SABINE,
Secretary.

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* Presented at the Fifty-fourth Annual Meeting of the Society, June 18-22, 1951.

[1] See p. 1258.

REPORT OF COMMITTEE C-21

ON

CERAMIC WHITEWARES*

In 1950, Committee C-21 held two meetings: on March 1 and September 21. In addition, the Executive Subcommittee met on April 23 and September 21. The present voting membership is 79, of whom 45 are producers, 13 are consumers, and 21 are general interest. The committee organization comprises subcommittees on Nomenclature, Tests and Specifications, and Research, respectively.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Nomenclature (A. S. Watts, chairman).—This subcommittee has a membership of 28 and holds monthly meetings except in July and August. It is now considering revisions of ten tentative definitions, at present given under the Tentative Definitions of Terms Relating To Ceramic Whitewares (C 242 - 50 T), as well as fourteen new definitions.

Subcommittee II on Tests and Specifications (F. P. Hall, chairman).—This subcommittee is divided into the following working groups:

A1 on Clays (T. A. Klinefelter, chairman).—This group held one meeting in 1950 and now has under consideration specifications for (*a*) sieve analysis and water content, (*b*) chemical analysis, (*c*) sampling, and (*d*) shrinkage.

A2 on Nonplastics (R. F. Sherwood, chairman).—This group is considering a rapid, and an umpire, method for chemical analysis of feldspar, flint, talc, pyrophyllite, and nepheline syenite. The method for particle size determination by the sieve method given in Department of Commerce Commercial Standard CS 23–30 also is being considered.

B on Processing Controls (C. H. Commons, chairman).—A questionnaire has been submitted to members of the whiteware industry for information on control testing and specifications now in industrial use.

C on Product Tests and Specifications (W. C. Mohr, chairman).—This group has ready for letter ballot, within the subcommittee, proposed test methods for: (*a*) true specific gravity, (*b*) linear thermal expansion, and (*c*) modulus of rupture of dry-pressed products. Also, a round-robin absorption test has been completed.

Subcommittee III on Research (C. G. Harman, chairman).—Ten members of this group cooperated in literature surveys to be used as the basis for committee work on various test methods. The reports of these surveys have been made available to members of Committee C-21.

This report has been submitted to letter ballot by the 79 voting members; 50 members returned their ballots, of whom 49 have voted affirmatively and 1 negatively.

Respectfully submitted on behalf of the committee,

J. H. KOENIG,
Chairman.

R. F. GELLER,
Secretary.

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* **Presented at the Fifty-fourth Annual Meeting of the Society, June 18–22, 1951.**

REPORT OF COMMITTEE C-22

ON

PORCELAIN ENAMEL*

Committee C-22 on Porcelain Enamel held two meetings during the year: one on October 12 and 13, 1950, as guests of the Westinghouse Electric Corp., Mansfield, Ohio, and one on March 6, 1951, in Cincinnati, Ohio, as a part of the Spring Committee Week.

During the past year three members were added to the committee and there were four removals. At the present time, the committee consists of 38 members, of whom 33 are voting members, with 18 classified as producers, 4 as consumers, and 11 as general interest.

NEW TENTATIVES

Subsequent to the Annual Meeting, Committee C-22 will submit to the Administrative Committee on Standards the following methods and definitions for acceptance as tentative:[1]

Tentative Methods of Test for:

Resistance of Porcelain Enameled Utensils to Boiling Acid,

Impact Resistance of Porcelain Enameled Utensils,

Sieve Analysis of Wet Milled and Dry Milled Porcelain Enamel,

Acid Resistance of Porcelain Enamels (Room Temperature Test).

Tentative Definitions of:

Terms Relating to Porcelain Enamel.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Research (G. H. McIntyre, chairman).—Surveys have been completed and are reported in papers carrying the following titles:

"Tentative List of Properties of Raw Materials and Finished Porcelain-Enameled Products" by E. E. Howe.

"A Review of Classifications of Various Types of Porcelain Enamels and Ceramic and Ceramic-Metal Coatings, Including Those for Use at High Temperatures" by Dwight G. Bennett, William Irby, and John T. Roberts.

"Gas Evolution Effects Associated with Steel, Enamel, and Enamel Processing" by B. J. Sweo.

"Review of Tests for the Estimation and Measurement of the Adherence of Porcelain Enamels and Ceramic Coatings to Iron, Steel, and other Metals" by L. S. O'Bannon.

In addition to the above reports, a fifth paper is being prepared for publication by E. H. Shands and J. J. Canfield on "Steel Surface on Fabricated Parts and Its Effect on Application of Porcelain Enamel." The subcommittee will form a task group to assemble, evaluate and correlate thermal shock data.

Subcommittee II on Nomenclature (F. A. Petersen, chairman) has recommended to Committee C-22 the publication of the Enamel Glossary as published by the American Ceramic Society (which consists of definitions of terms relating to porcelain enamel) as a tentative. The subcommittee is preparing for the use of the committee a compilation of trade names of proprietary products widely used in the industry.

Subcommittee III on Tests and Specifications (R. F. Bisbee, chairman):

Section 1, Raw Materials (W. A. Deringer, chairman) and Section 2, Materials in Process (S. E. Hemsteger, chairman) have completed the preparation of, and recommended for publication, a tentative sieve test for wet milled and dry milled porcelain enamel. Additional investigations are being carried on concerning rapid sieve testing methods of satisfactory reproducibility and the particle size analysis of sub-sieve fractions of milled porcelain enamels.

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* Presented at the Fifty-fourth Annual Meeting of the Society, June 18–22, 1951.

[1] See Editorial Note, p. 277.

Considerable progress has been made in work looking towards the preparation of tentatives concerning fusability of enamel frits, consistency of enamel slip, and the evaluation of enameling iron. A study is being made on the effect of the composition of water used in the milling of porcelain enamels upon the characteristics of the resultant porcelain enamel slip.

Section 3, Finished Products (J. H. Gregory, chairman) has submitted three proposed tentatives to Committee C-22 for acceptance, as noted earlier in this report. These tentatives include tests for: Acid Resistance of Porcelain Enamels (Room Temperature Test), Resistance of Porcelain Enameled Utensils to Boiling Acid, and Impact Resistance of Porcelain Enameled Utensils. The first test mentioned is also a standard test of the Porcelain Enamel Inst. while the latter two tests are standard tests of the Enameled Utensil Manufacturers Council. All three tests are widely used in the porcelain enamel industry.

Tests for warpage, resistance to abrasion, and reflectance of porcelain enamel coatings are under preparation.

This report was submitted to letter ballot of the committee which consists of 33 voting members; 30 members returned their ballots, of whom 29 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

W. N. HARRISON,
Chairman.

G. H. SPENCER-STRONG,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee C-22 presented to the Society through the Administrative Committee on Standards the following recommendations:

Tentative Definitions of:
Terms Relating to Porcelain Enamel (C 286 – 51 T).

Tentative Method of:
Test for Acid Resistance of Porcelain Enamels (C 282 – 51 T),
Test for Resistance to Boiling Acid of Porcelain Enameled Utensils (C 283 – 51 T),
Test for Impact Resistance of Porcelain Enameled Utensils (C 284 – 51 T), and
Test for Sieve Analysis of Wet Milled and Dry Milled Porcelain Enamel (C 285 – 51 T).

These recommendations were accepted by the Standards Committee on September 12, 1951, and the definitions and methods of test appear in the 1951 Supplement to Book of ASTM Standards, Part 3.

REPORT OF COMMITTEE D-1 ON PAINT, VARNISH, LACQUER, AND RELATED PRODUCTS*

Committee D-1 on Paint, Varnish, Lacquer, and Related Products held two meetings during the year: in Atlantic City, N. J., June 26 to 28, 1950, in connection with the 1950 Annual Meeting of the Society, and in Washington, D. C., February 26 to 28, 1951, in connection with the Spring Group Committee Meetings.

At the June, 1950, meeting, the following two papers were presented: "Adhesion Studies," by A. M. Malloy, U. S. Navy, Bureau of Aeronautics, and "Film Thickness Measurements,"[1] by E. J. Dunn, Jr., National Lead Co.

At the February, 1951, meeting, a panel discussion entitled, "So You Want to Measure Gloss!"[2] was held. Harry K. Hammond, III, served as moderator. The topics covered were: "Classification of Painted Surface According to Gloss; the 60-deg. Technique," by William Kiernan; "Solution to the High-Gloss Problem: Haze," by Mark Morse; and "Solution to the Low-Gloss Problem: Sheen," by Sam Huey.

Mr. R. J. Wirshing will present a paper on Weathering of Paints at the June meeting.

The year 1952 will mark the 50th Anniversary of the founding of Committee D-1. A committee of five will develop plans for presenting a special program and other events at the spring or June, 1952, meeting.

Percy H. Walker, a long-time member of Committee D-1, was given the ASTM Award of Merit by the Society at the June, 1950, Annual Meeting.

The Joint Committee of the Federation of Paint and Varnish Production Clubs, under the chairmansip of M. Van Loo, has approved five ASTM methods as follows: D 280–33 (Fed. 34–50) Method of Test for Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments; D 714–45 (Fed. 36–51) Method for Evaluating Resistance to Blistering of Paints on Metal when Subjected to Immersion or Other Exposure to Moisture or Liquids; D 555–50 T (Fed. 37–51) Methods of Test for Acetone Number of Heat-Bodied Drying Oils; D 967–48 T (Fed. 38–51) Method of Test for Heat Bodying Rate of Drying Oils; D 1012–49 T (Fed. 39–51) Method of Test for Aniline Point and Mixed Aniline Point of Hydrocarbon Solvents. Single and Multiple Panel Forms for Recording Results of Exposure Tests of Paint, D 1150–51 T (Fed. 35–50), which represents a revision of the earlier Federation form, have now been approved by both organizations. The Federation is now reviewing five ASTM methods and thirteen specifications.

The above approvals bring the total to 37 methods and specifications which have been jointly adopted by the Federation and ASTM. The Ford Viscosity Cup Method is also being considered jointly by the two groups.

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* Presented at the Fifty-fourth Annual Meeting of the Society, June 18–22, 1951.

[1] ASTM BULLETIN, No. 172, February, 1951, p. 35.

[2] To be abstracted in ASTM BULLETIN.

The Technical Coordinating Committee for the Paint Industry, on which Committee D-1 is represented, recently completed a survey among the technical men of the industry and obtained suggestions on technical problems which should receive the immediate attention of organizations represented on the Coordinating Committee and also other groups conducting scientific investigations in the field of protective coatings.

Committee D-1 is cooperating with Committees D-2 on Petroleum Products and Lubricants, and D-16 on Industrial Aromatic Hydrocarbons, on a review of distillation methods developed by these three committees.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1950 Annual Meeting, Committee D-1 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative:

Single and Multiple Panel Forms for Recording Results of Exposure Tests of Paints (D 1150 - 51 T).

Revision of Tentative Method of:

Producing Films of Uniform Thickness of Paint, Varnish, Lacquer and Related Products on Test Panels (D 823 - 47 T),

Test for Changes in Protective Coatings of Paint, Varnish, Lacquer and Related Products on Steel Surfaces When Subjected to Immersion (D 870 - 46 T), and

Conducting Exterior Exposure Tests of Paints on Wood (D 1006 - 49 T).

Revision of Tentative Specifications for:

Venetian Red (D 767 - 50 T).

These recommendations were accepted by the Standards Committee on March 2, 1951, with the exception of the last recommendation, which was accepted on May 12, 1951.

The new and revised tentatives appear in the 1951 Supplement to Book of ASTM Standards, Part 4.

PROPOSED METHODS TO BE PUBLISHED AS INFORMATION

Committee D-1 recommends that the following two proposed methods of test be published as information, as appended hereto:

Proposed Method of Test for:

20-Deg. Specular Gloss, and
85-Deg. Sheen.

NEW TENTATIVES

The committee recommends for publication as tentative the following two specifications and three methods of test as appended hereto.[3]

Specifications for:

Methanol (Methyl Alcohol),
Methyl Isobutyl Ketone.

Methods of Test for:

Purity of Acetone and Methyl Ethyl Ketone (Aqueous Hydroxylamine Method),

Roundness Determination of Glass Spheres, and

Total Chlorine in Polyvinyl Chloride Polymers and Copolymers Used for Surface Coatings.

REVISIONS OF TENTATIVES

Committee D-1 recommends that the following three tentatives be revised as indicated:

Tentative Specifications for Wood to be Used as Panels in Weathering Tests of Paints and Varnishes (**D 358 - 48 T**).[4]—Make the following changes:

Section 1.—Change the fourth sentence to read as follows by the addition of the italicized words:

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[3] These new tentatives were accepted by the Society and appear in the 1951 Supplement to Book of ASTM Standards, Part 4.

[4] 1949 Book of ASTM Standards, Part 4.

> Adequate evaluation of paint for wood requires testing on at least three woods, such as cedar or redwood, white pine *or ponderosa pine*, and southern yellow pine.

In the fifth sentence delete the words "therefore, they tend to give high estimates of paint durability," which refers to cedar and redwood, and delete "therefore, they tend to give a low estimate of paint durability," which refers to southern yellow pine.

Revise the last sentence of Section 1 to read as follows by the addition of the italicized words: "White pine *and ponderosa pine* stand in an intermediate position."

Section 6.—Revise the section heading to read "White Pine and Ponderosa Pine." Replace the present Section 6 by the following:

> 6. White pine wood shall be northern white pine (*Pinus strobus*), Western white pine (*Pinus monticola*), or sugar pine (*Pinus lambertiana*). Western white pine is sometimes called Idaho white pine. Ponderosa pine wood shall be *Pinus ponderosa.*

Tentative Specification for Dehydrated Castor Oil (**D 961 - 48 T**).[4]—Revise as follows:

Section 2.—Revise the table of requirement to read as follows:

	Unbodied		Bodied	
	Min	Max	Min	Max
Viscosity at 25 C.....	F	I	Z2	Z4
Specific Gravity, 15.5/15.5 C.........	0.930	0.941	0.948	0.970
Acid number.........	...	6	...	6
Saponification number...............	188	195	188	195
Iodine number (Wijs)	125	145	100	...
Color (Gardner 1933), No...............	...	6	...	7
Gel time at 600 F, min.	...	145	...	63
Set-to-touch time, hr.	...	2.5	...	1.4
Refractive Index at 25 C...............	1.4805	1.4825	1.4860	1.4890

Section 3(d).—In the first sentence change "reducing the viscosity to 1 poise" to read "reducing the viscosity to 1 stoke."

Tentative Method of Conducting Exterior Exposure Tests of Paints on Wood (**D 1006 - 49 T**).[4]—Revise as appended hereto.[5]

REVISION OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions in the following seven standards and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting so the revisions may be referred to letter ballot of the Society:

Standard Specifications for Spirits of Turpentine (**D 13 - 34**).[4]—In Table I on physical requirements change the specific gravity limits for Destructively Distilled Wood Turpentine to read "0.850 min," and "0.865 max."

Standard Definitions of Terms Relating to Paint, Varnish, Lacquer, and Related Products (**D 16 - 47**).[4]—Revise by the addition of the following new or revised definitions:

> *Water Paint.*—A paint, the vehicle of which is a water emulsion, water dispersion, or ingredients that react chemically with water.
>
> *Emulsion Paint.*—A paint, the vehicle of which is an emulsion of binder in water. The binder may be oil, oleoresinous varnish, resin, rubber or other emulsifiable binder. (New definition)
>
> *Ester Gum.*—A resin made from rosin and polyhydric alcohol, such as glycerin or pentaerythritol.
>
> *Penta Resin.*—Ester gum made from rosin and pentaerythritol. (New definition)
>
> *Thinner.*—Volatile organic liquid used to adjust consistency or to modify other properties of paint, varnish, and lacquer.
>
> *Spar Varnish.*—A varnish for exterior surfaces. The name originated from its use on spars of ships.
>
> *Sealer.*—A liquid composition to prevent excessive absorption of finish coats into porous surfaces; also a composition to prevent bleeding (see Size).
>
> *Primer.*—The first of two or more coats of a paint, varnish, or lacquer system.

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[5] The revised tentative method was accepted by the Society and appears in the 1951 Supplement to Book of ASTM Standards, Part 4.

Size.—Usually a liquid composition to prevent excessive absorption of all paints into plaster, old wall paint, and similar porous surfaces; also a liquid composition used as a first coat on metal to improve adhesion at succeeding coats (latter usage is limited to the metal decorating industry).

Note.—The terms sealer and size are almost synonomous, but usage has established certain differences. A sealer is ordinarily a thin varnish or clear lacquer and is usually applied on wood and metal surfaces. Ordinary painter's size is a thin solution of glue, starch or other water-soluble substance and is usually applied on plaster surfaces, but size used in metal decorating is a thin varnish.

Standard Methods of Sampling and Testing Turpentine (**D 233 – 48**).[4]—Revise by adding two new sections for determining evaporation residue and acidity as appended hereto.[6]

Standard Specifications for Shellac Varnishes (**D 360 – 41**).[4]—The committee recommends the revision of this standard, as appended hereto.[6]

Standard Methods of Sampling and Testing Aluminum Powder and Paste (**D 480 – 48**).[4]—Make the following revisions:

Section 4(d).—In the table under Weight of Sample, g, for Type I, change the present "2" to read "1" for *A*, and change the present "1.5" to read "2" for *B*. For Type II, Class *C*, change the Weight of Sample, g, from the present "1.5" to read "3.0." Revise the Footnote *b* to read: "Class *A* aluminum pigment is known commercially as 'extra fine lining', class *B* as 'standard lining' and class *C* as 'standard fineness for special uses.'"

Section 5(a).—At the end of the second sentence add "using 3.75 g of type A powder, 6.25 g of type B powder, or 10 g of type C powder with 40 ml of vehicle."

Section 5(b).—Revise the phrase "using 10 g of type A paste and 6 g of type B paste" to read "using 6.25 g of type A paste, 10 g of type B paste or 15 g of type C paste with 40 ml of vehicle."

Section 6(a).—In the fourth sentence delete the remainder of the sentence after the words "on the sieve" and add "using a No. 325 (44-micron) sieve for types A and B powder and a No. 100 (149-micron) sieve for type B powder."

Section 6(b).—At the end of the sentence delete "all types of paste" and add "types A and B paste and a No. 100 (149-micron) sieve for type C paste."

Section 8.—In the sixth sentence delete the last part of the sentence after the words "proportions of" and add "6.25 g of type A paste, 10 g of type B paste, or 15 g of type C paste with 40 ml of varnish (Note 2)."

Standard Specifications for Zinc Dust (Metallic Zinc Powder) (**D 520 – 47**).[4]—In Table I on Requirements for Composition under type II change "0.2" to read "0.8" for coarse particles on a No. 200 sieve.

Standard Method of Test for Dry to No-Pick-Up Time of Traffic Paint (**D 711 – 48**).[4]—Revise the terminology of this method from "Dry to No-Pick-Up Time" to read "No-Pick-Up Time" throughout the method.

ADOPTION OF TENTATIVES AS STANDARD

Committee D-1 recommends that the following 12 tentatives be approved for reference to letter ballot of the Society for adoption as standard:

Tentative Specifications for:

Red and Brown Iron Oxide Pigments (D 84 – 48 T).

Tentative Methods of:

Chemical Analysis of Dry Cuprous Oxide and Copper Pigments (D 283 – 48 T),

Test for Acetone Extract in Black Pigments (D 305 – 48 T),

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[6] The revised standard was accepted by the Society and appears in the 1951 Supplement to Book of ASTM Standards, Part 4.

Chemical Analysis of Zinc Yellow Pigment (Zinc Chromate Yellow) (D 444 - 48 T),
Test for 60-deg. Specular Gloss of Paint Finishes (D 523 - 49 T), revised as appended hereto,[10]
Analysis of Diatomaceous Silica Pigment (D 719 - 47 T),
Evaluating Degree of Resistance of Traffic Paint to Chipping (D 913 - 47 T),
Test for Abrasion Resistance of Coatings of Paint, Varnish, Lacquer, and Related Products by the Falling Sand Method (D 968 - 48 T),
Testing Pure Para Red and Toluidene Red Pigments (D 970 - 48 T),
Measurements of Dry Film Thickness of Paint, Varnish, Lacquer, and Related Products (D 1005 - 49 T),
Test for Aniline Point and Mixed Aniline Point of Hydrocarbon Solvents (D 1012 - 49 T), and
Conducting Exterior Exposure Tests of Paints on Steel (D 1014 - 49 T).

REVISION OF STANDARD AND REVERSION TO TENTATIVE

The committee recommends the revision of Standard Methods of Chemical Analysis of White Pigments (D 34 - 47)[4] and its reversion to tentative status. The proposed revision, appended hereto,[15] replaces the procedure for analysis of zinc pigments in sections 17 to 28 of Methods D 34.

WITHDRAWAL OF STANDARD

The committee recommends the withdrawal of the Standard Specifications for Lead Titanate (**D 606 - 42**) as this pigment has not been produced for several years.

EDITORIAL CHANGES IN STANDARDS

The committee recommends the following editorial changes in three specifications for blue pigments:

Standard Specifications for Iron Blue (**D 261 - 47**).[4]—Change Section 4 to read as follows:

4. (*a*) *Identification.*—Section 4 of the Tentative Methods of Chemical Analysis of Blue Pigments (ASTM Designation: D 1135).

(*b*) *Extenders and Diluents.*—Sections 12 to 17 inclusive, of the Tentative Methods of Chemical Analysis of Blue Pigments (ASTM Designation: D 1135).

(*c*) *Total Matter Soluble in Water.*—Matter Soluble in Water shall be determined by either of the following methods, as mutually agreed upon by the purchaser and the seller.

(*1*) Water-Soluble Matter by Extraction.—Section 9 of the Tentative Methods of Chemical Analysis of Blue Pigments (ASTM Designation: D 1135).

(*2*) Water-Soluble Salts by Electrical Conductivity.—Sections 10 and 11 of the Tentative Methods of Chemical Analysis of Blue Pigments (ASTM Designation: D 1135).

(*d*) *Moisture in Dry Pigment.*—Moisture shall be determined by either of the following methods, as mutually agreed upon by the purchaser and the seller:

(*1*) *Brabender Moisture Tester.*—Sections 5 and 6 of the Tentative Methods of Chemical Analysis of Blue Pigments (ASTM Designation: D 1135).

(*2*) *Toluene Distillation.*—Sections 7 and 8 of the Tentative Methods of Chemical Analysis of Blue Pigments (ASTM Designation: D 1135).

(*e*) *Organic Colors or Lakes.*—Section 18 of the Tentative Methods of Chemical Analysis of Blue Pigments (ASTM Designation: D 1135).

(*f*) *Pigment, Linseed Oil, and Moisture and Other Volatile Matter in Paste in Oil.*—Tests shall be conducted as specified for white linseed oil paints in Standard Methods of Chemical Analysis of White Linseed Oil Paints (ASTM Designation: D 215).

(*g*) *Mass Color and Tinting Strength.*—Standard Method of Test for Mass Color and Tinting Strength of Color Pigments (ASTM Designation: D 387).

Standard Specifications for Ultramarine Blue (**D 262 - 47**).[4]—Change Section 4 to read as follows:

4. (*a*) *Identification.*—Section 25 of the Tentative Methods of Chemical Analysis of Blue Pigments (ASTM Designation: D 1135).

(*b*) *Matter Soluble in Water.*—Section 27 of the Tentative Methods of Chemical Analysis of Blue Pigments (ASTM Designation: D 1135).

(*c*) *Moisture and Other Volatile Matter in Dry Pigment.*—Method A of the Standard Method of Test for Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments (ASTM Designation: D 280).

(*d*) *Coarse Particles.*—Standard Methods of Test for Coarse Particles in Pigments, Pastes, and Paints (ASTM Designation: D 185).

(*e*) *Organic Colors or Lakes.*—Sections 28 and 29 of the Tentative Methods of Chemical Analysis of Blue Pigments (ASTM Designation: D 1135).

(*f*) *Mass Color and Tinting Strength.*—Standard Method of Test for Mass Color and Tinting Strength of Color Pigments (ASTM Designation: D 387).

(*g*) *Pigment, Linseed Oil, and Moisture and Other Volatile Matter in Paste in Oil.*—Standard Methods of Chemical Analysis of White Linseed Oil Paints (ASTM Designation: D 215).

Standard Specifications for Copper Phthalocyanine Blue (**D 963 – 49**).[4]—Change Section 4(*d*) to (*h*) to read as follows:

(*d*) *Identification.*—Section 19 of the Tentative Methods of Chemical Analysis of Blue Pigments (ASTM Designation: D 1135).

(*e*) *Basic Dye Derivatives.*—Section 21 of the Tentative Methods of Chemical Analysis of Blue Pigments (ASTM Designation: D 1135).

(*f*) *Other Organic Coloring Matter.*—Section 22 of the Tentative Methods of Chemical Analysis of Blue Pigments (ASTM Designation: D 1135).

(*g*) *Ultramarine Blue.*—Section 23 of the Tentative Methods of Chemical Analysis of Blue Pigments (ASTM Designation: D 1135).

(*h*) *Iron Blue.*—Section 24 of the Tentative Methods of Chemical Analysis of Blue Pigments (ASTM Designation: D 1135).

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends that the following tentatives be continued as tentative without revision:

Specifications for:

Heavy Petroleum Spirits (Heavy Mineral Spirits) (D 965 – 48 T).

Methods of:

Laboratory Test for Degree or Resistance of Traffic Paint to Bleeding (D 969 – 48 T).

Testing Bituminous Emulsions for Use as Protective Coatings for Metal (D 1010 – 49 T), and

Test for Night Visibility of Traffic Paints (D 1011 – 49 T).

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[7]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II on Drying Oils (Francis Scofield, chairman) has completed an extensive revision of the Standard Methods for Testing Drying Oils (D 555 – 47). The revised methods will replace the Tentative Methods of Testing Drying Oils (D 555 – 50 T) and the Tentative Method of Test for Heat Bodying Rate of Drying Oils (D 967 – 48 T). The revised methods cover all of the present test procedures in the three existing standards and in addition will include a new test for diene value and set-to-touch time. The revised methods will be submitted to the Administrative Committee on Standards subsequent to the Annual Meeting.[7a]

Subcommittee III on Bituminous Emulsions (R. H. Cubberly, chairman) has recommended the continuance as tentative of Method D 1010 – 49 T, Testing Bituminuous Emulsions for Use as Protective Coatings on Metal, as additional revisions are contemplated in this method. The committee is also drafting proposed Tentative Specifications for Coal-Tar Base Emulsions for use as Protective Coatings on Metal, and also has prepared a Tentative Specification for Asphalt-Base Emulsions for use as Protective Coatings for Metal.[7a]

Subcommittee IV on Traffic Paints (W. G. Vannoy, chairman) prepared the proposed Tentative Method of Test for Roundness Determination of Glass Spheres, appended hereto,[8] and is developing a method of test for crushing resistance determination of glass spheres. Cooperative work is being continued on: a method of test for dirt retention time, accelerated durability tests, an accelerated suspension test, and clarity of glass beads. Work is planned on development of a method of test for evaluating wetting characteristics of glass beads.

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[7] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

[7a] See Editorial Note, p. 286.

[8] The new tentative was accepted by the Society and appears in the 1951 Supplement to Book of ASTM Standards, Part 4.

Subcommittee V on Volatile Solvents for Organic Protective Coatings (M. B. Chittick, chairman) prepared an additional test covering evaporation residue and a test for acidity to be added to the Standard Methods of Sampling and Testing Turpentine (D 233 – 48), as mentioned earlier in this report. The subcommittee is continuing cooperation with Research Division X of Committee D-2 on copper strip corrosion, as well as further work on solvency and distillation.

Subcommittee VI on Definitions (C. H. Rose, chairman) has prepared new definitions for "Penta Resin" and "Emulsion Paint", and revised definitions for "Water Paint," "Ester Gum," "Spar Varnish," "Primer," "Sealer," and "Size." The subcommittee is also preparing definitions for about thirty terms for pigments, lacquers, alkyd resins, and solvents.

Subcommittee VII on Accelerated Tests (R. H. Sawyer, chairman) prepared the revision of the method of Immersion Testing (D 870 – 46 T) and Specifications for Wood to be Used as Panels in Weathering Test of Paints and Varnishes (D 358 – 48 T). Cooperative work is being conducted on the development of a method of rating blistering and accelerated weathering machines.

Subcommittee VIII on Chemical Analysis (C. Y. Hopkins, chairman) is preparing a compilation of tentative methods of test for common properties of certain pigments. A comparative study is being undertaken of two proposed methods for determining the TiO_2 content of titanium-base pigments. The subcommittee also prepared the extensive revisions of Chemical Analysis of White Pigments (D 34 – 47).

Subcommittee IX on Varnishes (J. C. Weaver, chairman) is preparing a Method of Test for Viscosity of Paints, Varnishes, and Lacquers by the Ford Viscosity Cup. A survey of color ranges actually encountered in practical clear vehicles for coatings is being conducted. Cooperative tests are being undertaken to define more carefully the raw linseed oil used in miscibility tests. Work is being continued on the development of a rapid method for the determination of nonvolatile matter by use of a thermostatically controlled hot plate as a heat source. The proposed method of testing alkali resistance is under study. It is expected that by June, 1951, all results of exterior exposures by seven collaborators of six different varnishes will be reported, and that a section on methods of application, exposure, and reading of exposure of varnish will be recommended for incorporation in Methods D 154. Foster D. Snell Laboratories is cooperating with the subcommittee on the tests of the wearability of floor varnishes.

Subcommittee X on Optical Properties (W. C. Granville, chairman) prepared the revision of the Method for Mass Color and Tinting Strength of Color Pigments (D 387–36). It also sponsored a paper, "Method of Test for Hiding Power of Paints," by Marshall Switzer.[9] Certain revisions in Method D 523–49 T were prepared and the revised tentative recommended for adoption as standard.

Subcommittee XI on Resins (C. F. Pickett, chairman) prepared the method for the total chlorine in polyvinyl chloride polymers used for surface coatings, which is recommended for publication as tentative as mentioned earlier in this report. A method for the determination of softening points has been developed and is being submitted to Committee E-1. A method for the determination of free phenol in phenolic resins and solutions used for coatings has also been developed.

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[9] Published in the July ASTM BULLETIN, No. 175, July, p. 68 (TP 158).

Round-robin tests are in progress on nonvolatile determinations by precipitation and foil methods, resin solutions, and viscosities, determination of phthalic anhydride in alkyd resins, and solvent tolerance of amino resins.

Surveys are in progress on color of resins and resin solutions, qualitative test for phenols, and qualitative and quantitative test methods for bis-phenol, and tests for vinyl polymers.

Subcommittee XII on Exterior Exposure Testing of Paints on Wood (W. R. Fuller, chairman) prepared single panel record form and multi-panel paint inspection sheet, and these have been adopted by both the ASTM and the Federation of Paint and Varnish Production Clubs under ASTM Designation D 1150–51 T, and Federation Standard No. 35. The subcommittee also prepared revision of Tentative Method of Conducting Exterior Exposure Tests of Paints on Wood (D 1006–49T), mentioned earlier in this report.

Subcommittee XIII on Shellac (C. C. Hartman, chairman) prepared the revisions of the Standard Specifications for Shellac Varnishes (D 360–41) which are recommended for immediate adoption.

Subcommittee XIV on Electrometric Testing of Paint Films (D. L. Hawke, chairman) is developing a method of time-potential data. A review of published work is being conducted.

Subcommittee XV on Pigments (C. L. Crockett, chairman) prepared the revisions of specifications for venetian red, metallic zinc powder, copper phthalocyanine blue, iron blue, and ultramarine blue. Work is progressing on the terminology of reporting the fineness of particle distribution.

Subcommittee XVI on Printing Inks (M. C. Rogers, chairman).—Seven groups are making round-robin tests on methods of test for printing inks.

Subcommittee XVII on Flash Point (A. L. Brown, chairman).—Steps are being taken to distribute two proposed modifications of the Tag open cup test, namely, Manufacturing Chemists Assn. and Dean versions, secure collaborators, and run tests on selected materials. Operators are requested to submit their results and comments on difficulties encountered.

Subcommittee XVIII on Physical Properties (M. R. Euverard, chairman) has recommended that the Tentative Methods for Producing Films of Uniform Thickness of Paint, Varnish, Lacquer, and Related Products on Test Panels (D 823–51 T) be retained as tentative, and that the Tentative Method of Test for Abrasion Resistance of Coatings of Paint, Varnish, Lacquer, and Related Products by the Falling Sand Method (D 968–48 T) and the Tentative Method for Measurement of Dry Film Thickness of Paint, Varnish, Lacquer, and Related Products (D 1005–49 T) be adopted as standard.

Subcommittee XXV on Cellulose and Related Products (F. H. Lang, chairman) prepared the Specifications for Methanol, Methyl Isobutyl Ketone, and Method of Test for Purity of Acetone and Methyl Ethyl Ketone, all of which are appended to this report.[8] The subcommittee has reported progress on the revision of the Methods of Testing Nitrocellulose, Clear Lacquers, and Lacquer Enamels (D 333 – 40).

Subcommittee XXIX on Painting of Metals (A. J. Eickhoff, chairman) is preparing a set of photographic standards to be representative of various conditions of ferrous surfaces prior to painting. This subcommittee recommends the adoption as standard of the Tentative Method for Conducting Exterior Exposure Tests of Paints on Steel (D 1014 – 49 T) which has been tentative for two years.

This report has been submitted to letter ballot vote of the committee, which consists of 342 voting members; 144 members returned their ballots, of whom 141 have voted affirmatively and 3 negatively.

Respectfully submitted on behalf of the committee,

W. T. PEARCE,
Chairman.

C. H. ROSE,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-1 presented to the Society through the Administrative Committee on Standards the following recommendations:

Tentative Method for:
- Measurement of Dry Film Thickness of Non-Magnetic Coatings of Paint, Varnish, Lacquer and Related Products Applied on a Magnetic Base (D 1186 – 51 T).

Tentative Specifications for:
- Asphalt-Base Emulsions for Use as Protective Coatings for Metal (D 1187 – 51 T).

These recommendations were accepted by the Standards Committee on November 26, 1951, and the method and specifications are available as separate reprints.

On December 13, 1951, the Administrative Committee on Standards accepted the following recommendations submitted by Committee D-1:

Tentative Methods of:
- Testing Drying Oils (D 555 – 51 T).

Withdrawal of Standard Methods of:
- Testing Drying Oils (D 555 – 47).

Withdrawal of Tentative Methods of:
- Testing Drying Oils (D 555 – 50 T), and
- Test for Heat Bodying Rate of Drying Oils (D 967 – 48 T).

The new tentative methods D 555 – 51 T appear in the 1951 Supplement to Book of ASTM Standards, Part 4.

APPENDIX I

PROPOSED METHOD OF TEST FOR 20-DEG SPECULAR GLOSS[1,2]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

Scope

1. This method of test[3] is intended to supplement the Tentative Method of Test for 60-deg Specular Gloss (ASTM Designation: D 523)[4] by providing a more sensitive means for differentiating essentially plane, high-gloss paint specimens. It is particularly useful for specimens having a gloss value of more than 70 as determined by Method D 523.

Definition

2. *20-Deg Specular Gloss*, for the purpose of this method, is defined as the 20-deg specular reflectance of a sample relative to a plane, polished surface having an index of refraction of 1.540. This surface is assigned a value of 100.0. Specular reflectance measurements are to be made with apparatus having the illuminating and viewing beams specified in Section 3.

Apparatus

3. (*a*) The apparatus shall consist of a stable light source and lens furnishing an incident beam of light with its axis 20 deg from the perpendicular to the specimen, a means for positioning the specimen, and a receptor to receive light reflected in the region around the angle of specular (mirror) reflection. A receptor lens may or may not be employed, but in any case a sharp image of the source shall be formed in the center of the receptor field stop (entrance window).

(*b*) The source and receptor apertures shall be rectangular and shall conform to the following specifications:

Apertures	In Plane of Measurement	Perpendicular to Plane of Measurement
Source	1.00 deg max.	2.80 deg max.
Receiver ...	1.80 ± 0.05 deg	3.30 ± 0.10 deg

(*c*) There shall be no vignetting or interception by stops or diaphragms of rays supposed to register. The source-receptor combination employed shall give readings substantially the same as those obtained if the source were the standard illuminant *C* (average daylight) recommended by the International Commission on Illumination (ICI) and the receptor response were the same as that of the luminosity function, $\bar{y}$, of the ICI standard observer. The photometric portion of the apparatus shall give readings directly proportional to the light flux passing through the receptor entrance window, accurate to ±1.

Reference Standard

4. The primary working standard shall be a piece of polished black glass of refractive index 1.540, assigned a 20-deg

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[1] This proposed method is under the jurisdiction of the ASTM Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Published as information, June, 1951.

[3] For additional information related to this method, reference may be made to the paper by S. C. Horning and M. P. Morse, "The Measurement of Gloss of Paint Panels," *Official Digest*, Federation of Paint and Varnish Production Clubs, March, 1947, p. 153.

[4] 1951 Supplement to Book of ASTM Standards, Part 4.

specular gloss value of 100.0. Black glass of different refractive index may be used by increasing or decreasing the gloss setting of the instrument 0.3 for each 0.001 the index is above or below 1.540. For example, if the index of the standard is 1.530, the instrument should be set to read 97.

Specimen Preparation

5. Specimens may be prepared by spraying or doctor-blading on plate glass or flat metal panels, keeping spray wave to a minimum. If it is desired to determine the maximum gloss obtainable or to make the most critical differentiation between paints, doctor-blading on plate glass is recommended.

Preparation and Standardization of Apparatus

6. (*a*) Adjust the instrument, according to the manufacturer's instructions, to read the assigned value of the polished black glass standard.

(*b*) Read the gloss of a medium "high-gloss" standard and a low "high-gloss" standard.[5] If the readings for the latter two standards differ from their assigned values by more than 5 per cent of the gloss value, the instrument should be readjusted according to manufacturer's instructions or returned to the manufacturer for adjustment.

Method of Measurement

7. (*a*) Operate the glossmeter according to instructions of the manufacturer for power supply, warm-up period, dust-free atmosphere, temperature, humidity, darkness of surroundings, and handling of specimens.

(*b*) Make settings on the working standard at the start and completion of every period of glossmeter operation, and at intervals during this operation sufficiently frequent to ensure that the instrument response is practically constant.

(*c*) Measure at least three portions of the surface of the test specimen to give an indication of the degree of surface uniformity, and to provide an average that is typical of the surface.

Report

8. Report the average of three readings as 20-deg specular gloss according to this method, giving the maximum deviations obtained above and below the average.

Uncertainty of Results

9. (*a*) For the same surface area of a flat specimen, a properly designed instrument can be expected to give a standard deviation of 0.5 gloss unit for five consecutive readings.

(*b*) For the same surface area of a flat specimen, different instruments properly calibrated can be expected to give a standard deviation of 1.5 gloss units.

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[5] Standards calibrated for use with this method may be obtained from the National Bureau of Standards, Washington 25, D. C.

APPENDIX II

PROPOSED METHOD OF TEST FOR 85-DEG SHEEN[1,2]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race St., Phildelphia, Pa.

Scope

1. This method of test is intended for classifying paint finishes according to sheen (Note), that is, specular gloss at near-grazing angles. The method may also be used for intercomparison of the sheen of finishes which, while being generally similar in appearance at near-grazing angles, exhibit small differences in sheen. This method has been proved useful for finishes that have low gloss; its usefulness for higher-gloss finishes has not been established.

NOTE.—This method of test for sheen is intended to supplement the Tentative Method of Test for 60-Deg Specular Gloss of Paint Finishes (ASTM Designation: D 523),[3] and should not be considered an alternative. There seems to be no simple correlation between sheen at 85 deg and specular gloss at smaller angles; a finish having low specular gloss at smaller angles may or may not have low sheen.

Definition

2. Sheen, for the purpose of this method, is defined as 85-deg specular reflectance measured with the geometric conditions specified in Section 3(*b*), and based on a scale on which polished black glass, having an index of refraction of 1.532, is assigned a value of 100.

Apparatus.

3. (*a*) *General.*—The apparatus shall include a light source for furnishing a beam of rays incident on the specimen, means for reproducibly locating the surface of the specimen, and a receptor positioned to receive certain rays reflected by the specimen. There shall be included a measurement mechanism to translate the received light flux into readings on the sheen scale. The first-mentioned three parts, with or without the measurement mechanism, shall be mounted rigidly in a light-tight housing. The interior walls of this housing shall be black and sheen-free.

(*b*) *Geometric Conditions.*—The incident beam shall be substantially parallel, with its axis 85 ± 0.1 deg from the perpendicular to the test surface. The receptor shall be so located as to define a receptor beam axis, which is the mirror image of the axis of the incident beam. With a plane front-surface reflector in the specimen position, an image of the light source shall be formed substantially in the plane of the receptor window and centered in the window. The length of the illuminated area of the specimen shall be not more than one half the distance from the center of this area to the receptor window. The angular dimensions of

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[1] This proposed method is under the jurisdiction of the ASTM Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Published as information June, 1951.

[3] 1951 Supplement to Book of ASTM Standards, Part 4.

source and receiver (Note 1) shall be as follows:

	Aperture and Tolerance[a] (Degrees)	
	In Plane of Measurement	Perpendicular to Plane of Measurement
Source	no more than 1	no more than 3
Receiver	4.0 ± 0.3	6.0 ± 0.3

[a] These tolerances are so chosen so that variation in either source or receiver aperture will produce a change of no more than -- sheen units.

NOTE 1.—These angles are double those by which rays in the irradiator and receptor beams may spread from the principal ray of source and receptor beams. Angular size of the source is measured from the center of the source lens. Angular size of the receptor window is measured from the center of the receptor lens. The latter lens may be omitted if adjustments are made in the angular size of source and receptor so as to obtain equivalent sheen readings.

(*c*) *Vignetting*.—There shall be no loss of rays that should be registered by the receptor, according to the geometric requirements given in Paragraph (*b*), due to interception by stops, diaphragms or otherwise.

(*d*) *Spectral Conditions*.—The instrument shall give results not significantly different from those that would result if its light source were the standard illuminant *C* of the International Commission on Illumination (ICI), and the receptor had the spectral response of the ICI luminosity function (Note 2).

NOTE 2.—Specular reflection is in general spectrally nonselective; therefore in practice it has proved unnecessary to require close duplication of the specified spectral conditions. Measurements made with an incandescent lamp operated near its rated voltage and a barrier-layer type photocell do not require a filter for spectral correction.

(*e*) *Measurement Mechanism*.—The measurement mechanism, such as photocell and microammeter, shall give a numerical indication which is proportionate to the received light flux within ± 1 (Note 3).

NOTE 3.—The receptor sensitivity shall be substantially uniform over its surface or a light-diffusor shall be located in the receptor window or between it and the photocell.

Sheen Standards

4. The primary sheen standard shall be a piece of polished black glass having an index of refraction of 1.532 and an assigned value of 85 deg specular reflectance of 100 (Note). Working standards having intermediate sheen values are desirable, if they are calibrated against the primary standard on a sheenmeter known to conform accurately to the geometry requirements of the method. For the medium to low sheen range, such standards may be made of ground opaque glass or other material having hard, flat, uniform surface. Such standards should be checked from time to time by intercomparison, as contamination of surfaces by dirt or filmy deposits may cause unsuspected changes in sheen readings.

NOTE.—Accurate measurement of the index of refraction of the primary standard to three decimal places is not required, as a change in index of refraction from 1.5 to 1.6 will change the sheen scale reading by approximately one unit. A change in angle of irradiation of 0.1 deg from 85 deg, however, will change the sheen scale reading by one unit.

Preparation and Selection of Specimens

5. (*a*) The many factors which may affect the sheen of laboratory-prepared specimens will not be discussed here. When the test requires the preparation of specimens, the manufacturer's instructions for use of the material shall be carefully followed, or a particular technique of specimen preparation shall be agreed upon in advance by the laboratories concerned. It should be noted that in general, washing or rubbing will materially affect the sheen of a specimen.

(*b*) Specimens selected for measurement shall be as plane (flat) as it is possible to obtain them. Sheen readings taken on specimens with curved surfaces are subject to large error.

Method of Measurement

6. (*a*) The sheenmeter shall be operated according to the manufacturer's instructions.

(*b*) The instrument shall be calibrated by means of a working standard at sufficiently frequent intervals to insure constancy of response.

(*c*) At least three areas of the specimen surface shall be measured to provide an indication of uniformity and an average that is typical of the surface. Specimens shall be rotated 45 deg in their plane between readings, except specimens showing brush marks, in which case specimens shall be positioned with the marks parallel to the plane of measurement and rotated 180 deg between readings.

Diffuse Correction

7. No diffuse correction is required with sheen measurements. The diffuse reflectance contribution to a sheen reading is negligible.

NOTE.—The correction for a perfectly reflecting, perfectly diffusing specimen amounts to 0.05 sheen unit.

Report

8. (*a*) *Classification.*—Where sheen classification is desired, the specimens shall be reported as follows:

Instrument Sheen Reading	Report as
70 and above	High sheen
70 to 25, incl.	Medium sheen
25 to 9, excl.	Low sheen
9 and under	Sheen free

(*b*) *Numerical Rating.*—Where instrument readings are reported, they shall be given to the nearest unit when above 9 and to the nearest tenth unit when 9 and under. Specimens having areas differing in sheen from the average for the specimen by more than 10 per cent, shall be reported by average and deviation.

(*c*) *Other Data.*—Where test specimens have been prepared, the method of preparation shall be described or otherwise identified. The sheenmeter employed shall be identified by maker's name and model. Working standards used shall be identified.

Uncertainty of Results

9. The tolerances given in Section 3 for the angular size of source and receptor, for linearity of the measurement mechanism, and other factors, are estimated to provide a potential uncertainty of measurement for this method of about 10 per cent of the sheen reading. If working standards similar in sheen and surface character to the specimens being measured are exchanged by the interested laboratories, discrepancies in sheen readings can be reduced to a minimum.

REPORT OF COMMITTEE D-2

ON

PETROLEUM PRODUCTS AND LUBRICANTS[*]

In the interest of brevity, this report of Committee D-2 on Petroleum Products and Lubricants is confined to the recommendations of the committee. A separate report describing the activities of the committee during the year preceding the Annual Meeting in June, 1951, and the program of the committee for the year beginning in June, 1951, will be published in the ASTM BULLETIN.[1]

PROPOSED METHODS TO BE PUBLISHED AS INFORMATION

Technical Committees and Research Divisions of Committee D-2 have submitted seven proposed methods of test which are recommended for publication as information as appended to this report.

Appendix I. Method for Penetration of Lubricating Greases Worked More Than 60 Strokes. It is expected that this method will eventually be incorporated into ASTM Method D 217 – 48,[2] Test for Cone Penetration of Lubricating Grease.

Appendix II. Method for Measurement of Density and Specific Gravity of Liquids to the Fifth Place (Bingham Pycnometer Method).

Appendix III. Test for Saponification Number of Petroleum Products (Para-Xylenol Blue Color-Indicator Method).

Appendix IV. Test for Oil Content of Petroleum Waxes.

Appendix V. Test for Kinematic Viscosity.

Appendix VI. Test for Motor and Research Octane Numbers of Small Samples.

Appendix VII. Test for Motor and Research Octane Numbers of Small Samples by Multi-Pipet System.

The committee also recommends for publication, as information only in ASTM Method D 943, Test for Oxidation Characteristics of Inhibited Steam Turbine Oils, a proposed revision of Section 8 to read as follows:

8. *Precision.*—Results when expressed in hours to reach a neutralization value of 2.0 should not differ from the mean by more than the following amounts (based on 95 per cent probability level):

Repeatability, One Operator and Apparatus	Reproducibility,[a] Different Operators and Apparatus
12 per cent	37 per cent

[a] Average of two results from each operator.

NEW TENTATIVES

Committee D-2 recommends that the following four methods of test be accepted for publication as tentative:

Tentative Methods of Test for:[3]

Total Inhibitor Content (p-Tertiary Butylcatechol) of Butadiene. This method is an editorial revision of Method 2.1.9.1 in the Butadiene Laboratory Manual of the Office of Rubber Reserve. Section I of Technical Committee H has found that this method is in use and is reliable.

Bromine Number of Petroleum Distillates (Color Indicator Method) (see Appendix II, 1950 Report of Committee D-2).[4] The method has been further revised and is now recommended for publication as tentative with the following changes:

Section 1 (*b*).—Revise to read as follows: "(*b*) The magnitude of the bromine number is an indication of the quantity of bromine-reactive constituents, not an identification of constituents; therefore its application as a measure of olefinic unsaturation should not be undertaken without a study of the appended 'Reported Behavior of Compounds in the Color Indicator and Electrometric Bromine Number Methods.'"

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[*] Presented at the Fifty-fourth Annual Meeting of the Society, June 18–22, 1951.

[1] Published in the ASTM BULLETIN, No. 177, October, 1951, p. 21.

[2] 1949 Book of ASTM Standards, Part 5.

[3] These methods were accepted as tentative by the Society and appear in the 1951 Supplement to Book of ASTM Standards, Part 5.

[4] *Proceedings*, Am. Soc. Testing Mats., Vol. 50, p. 276 (1950).

Section 5 (c).—In the first sentence delete "(or the equivalent 44.1 g of NaBr and 12.58 g of $NaBrO_3$)." After the first sentence, add the following: "If the determinations of the bromine number of the reference olefins specified in Section 6 using this solution do not conform to the prescribed limits, or if for reasons of uncertainties in the quality of primary reagents, or procedural details, or if for reason of preference it is considered desirable to determine the normality of the solution, the solution shall be standardized and the determined normality used in subsequent calculations. The standardization procedure shall be as follows:"

Section 6.—In Note 3 change "194.8" to "194.6"; also change "142.3" to "142.4" in the two places where it appears in the note.

Section 9.—Revise the table on Precision by substituting the following:

ASTM 90 per cent Distillation Point, deg Fahr	Range of Bromine Number	Repeatability	Reproducibility
Under 400	0 to 1.5	0.2	0.4
	Over 1.5 to 20	0.5	1
	Over 20 to 50	1	2
	Over 50 to 100	2.5	5
400 to 620	0 to 1.5	0.2	0.4
	Over 1.5 to 10	2	4
	Over 10 to 20	3	6

Appendix.—Include in an Appendix the "Reported Behavior of Compounds in the Color Indicator and Electrometric Bromine Number Methods" which is appended[5] to this report.

Bromine Number of Petroleum Distillates (Electrometric Method) (see Appendix III, 1950 Report of Committee D-2).[6] The method has been further revised and is recommended for publication as tentative with the following changes:

Section 1 (b).—Change Paragraph (*b*) to read as follows: "(*b*) The magnitude of the bromine number is an indication of the quantity of bromine-reactive constituents, not an identification of constituents; therefore, its application as a measure of olefinic unsaturation should not be undertaken without a study of the appended 'Reported Behavior of Compounds in the Color Indicator and Electrometric Bromine Number Methods.'"

Section 4.—Add to Section 4 on Apparatus the following new item: "(*c*) *Iodine Number Flasks.*—500 ml, glass-stoppered, will be required."

Section 5 (b).—In the first sentence delete "in a volumetric flask" and add the following new paragraphs: "If the determinations of the bromine number of the reference olefins specified in Section 6 using this solution do not conform to the prescribed limits, or if for reasons of uncertainties in the quality of primary reagents, or procedural details, or if for reason of preference it is considered desirable to determine the normality of the solution, the solution shall be standardized and the determined normality used in subsequent calculations.

The standardization procedure shall be as follows: to standardize, place 50 ml of glacial acetic acid and 1 ml of HCl (sp gr 1.18) in a 500-ml iodine number flask. Chill the solution in an ice bath for approximately 10 min and, with constant swirling of the flask, add from a 10-ml calibrated buret 5 ± 0.01 ml of bromide-bromate solution at the rate of 1 or 2 drops per sec. Stopper the flask immediately, shake the contents, place it again in the ice bath, and add 5 ml of KI solution in the lip of the flask. After 5 min remove the flask from the ice bath and allow the KI solution to flow into the flask by slowly removing the stopper. Shake vigorously, add 100 ml of water in such a manner as to rinse the stopper, lip, and walls of the flask, and titrate promptly with $Na_2S_2O_3$ solution. Near the end of the titration, add 1 ml of starch indicator solution and titrate slowly to disappearance of the blue color. Calculate the normality of the bromide-bromate solution as follows:

$$\text{Normality of bromide-bromate solution} = \frac{\text{ml of } Na_2S_2O_3 \times \text{normality of } Na_2S_2O_3}{5}$$

Repeat the standardization until duplicate determinations do not differ from the mean by more than $\pm 0.002\ N$.

(*c*) *Potassium Iodide Solution* (150 g per l).

(*d*) *Sodium Thiosulfate Solution* (*Standard 0.1N*).—Dissolve 25 g of $Na_2S_2O_3 \cdot 5H_2O$ in water and add 0.01 g of Na_2CO_3 to stabilize the solution. Dilute to 1 liter and mix thoroughly by shaking. Standardize by any accepted procedure that determines the normality with an error not greater than ±0.0002. Restandardize at intervals frequent enough to detect changes of 0.0005 in normality.

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[5] This appendix was accepted by the Society and is appended to the new Tentative Method of Test for Bromine Number of Petroleum Distillates (Color Indicator Method) (D 1158 – 51 T) and the new Tentative Method of Test for Bromine Number of Petroleum Distillates (Electrometric Method) (D 1159 – 51 T) and appears in the 1951 Supplement to Book of ASTM Standards, Part 5.

[6] *Proceedings*, Am. Soc. Testing Mats., Vol. 50, p. 280 (1950).

(*e*) *Starch Solution.*—Titrate 5 g of arrowroot starch and 5 to 10 mg of HgI_2 with 3 to 5 ml of water. Add the suspension to 2 l of boiling water and boil for 5 to 10 min. Allow to cool and decant the clear, supernatant liquid into glass-stoppered bottles."

Section 6.—In Note 3 change "194.8" to "194.6"; also change "142.3" to "142.4".

Section 7.—Delete the second sentence. In Table II on Sample Size in this section replace the present values with the following:

Bromine Number	Sample Size, g
0 to 10	20 to 16
Over 10 to 20	10 to 8
Over 20 to 50	5 to 4
Over 50 to 100	2 to 1.5
Over 100	1 to 0.5

Section 9.—Revise the table on Precision in this section by replacing the present values by the following:

ASTM 90 per cent Distillation Point, deg Fahr	Range of Bromine Number	Repeatability	Reproducibility
Under 400	0 to 1.5	0.3	0.6
	Over 1.5 to 20	0.7	1.5
	Over 20 to 50	2	4
	Over 50 to 100	2.5	5
400 to 620	0 to 1.5	0.5	1
	Over 1.5 to 10	1	2
	Over 10 to 20	2	4

Appendix.—Include in an Appendix the "Reported Behavior of Compounds in the Color Indicator and Electrometric Bromine Number Methods" which is appended[5] to this report.

Reduced Pressure Distillation of Petroleum Products (see Appendix VII, 1950 Report of Committee D-2).[7] Minor revisions in the text and corrections in the illustrations have been incorporated into the revised method which is recommended for publication as tentative. Make the following revisions in the method:

Section 3 (*f*).—Revise to read as follows: "(*f*) *Pressure Measuring System* capable of measuring absolute pressure with an accuracy of 1 per cent of the observed reading in the range of 1 mm of mercury to atmospheric pressure. Devices which meet this requirement are discussed in the Appendix, Section A4 (*a*)."

Section 7 (*d*).—In the first sentence delete "minimum amount of."

Section 9.—Replace the present Section 9 on Precision with the following:

9. The following data should be used for judging the acceptability of results:

(*a*) *Repeatability.*—Duplicate results by the same operator should be considered suspect if they differ by more than the following amounts:

Pressure	Repeatability
1 mm of Mercury, Absolute:	
5 per cent recovered and greater	10 F
10 mm of Mercury, Absolute:	
5 per cent recovered and greater	8 F
50 mm of Mercury, and higher:	
5 per cent recovered and greater	10 F

(*b*) *Reproducibility.* The averages of duplicate results obtained in different laboratories should be considered suspect if they differ by more than the following amounts:

Pressure	Reproducibility
1 mm of Mercury, Absolute:	
5 per cent recovered	30 F
10 per cent recovered and greater	20 F
10 mm of Mercury, Absolute:	
5 per cent recovered	20 F
10 per cent recovered and greater	15 F
50 mm of Mercury, and higher:	
5 per cent recovered	30 F
10 per cent recovered and greater	20 F

Appendix.—In Section A4 on Pressure System, replace the present Paragraph (*a*) with the following new paragraphs:

A4. (*a*) *Pressure Measuring System.*—In the pressure range of 20 mm of mercury, absolute, and below, the use of a McLeod type gage is mandatory. The gage shall have an accurately known volume ratio of at least 10 to 1 and be of the quadratic scale type. It shall be designed so that in the range in which it is intended to be used it shall have an accuracy, sensitivity, and readibility of 1 per cent of the observed reading.

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[7] *Proceedings*, Am. Soc. Testing Mats., Vol. 50, p. 299 (1950).

NOTE 11.—While it is desirable that a single gage covering the range 1 to 20 mm of mercury and possessing the above characteristics be employed, it may be more practicable to select a gage of shorter range in order to obtain improved accuracy.

Above 20 mm a carefully constructed and filled closed-end mercury manometer may be used. Both arms of the manometer shall have the same inside diameter of not less than 10 mm, in order to avoid capillary and wetting effects. Filling shall be accomplished only after thorough cleaning and degassing of both the manometer and mercury. The manometer shall be equipped with means for accurately measuring the differences in level of the arms under operating conditions. In the range above 20 mm, other devices may be used provided the accuracy of 1 per cent of the observed reading can be met.

In any pressure measuring system adequate traps shall be provided to prevent vapors from the distillation apparatus from interfering with the functioning of the gages, or accidental flow of mercury or other manometric liquid into the distillation apparatus.

NOTE 12.—Pressure measuring devices may be checked in the laboratory by the use of a carefully constructed and operated comparison manometer as shown in Fig. 1. The essential parts of the system consist of: a manometer of sufficient length to cover the range of the instrument under test; forepump, diffusion pump, and traps to provide rapid evacuation to less than 1-micron pressure; a McLeod gage for checking the pressure on the reference or zero arm of the manometer; ballast chambers or bulbs; suitable connections to the instrument under test; and an accurate cathetometer to measure the difference in arm levels of the manometer. The ballast bulbs shall be at least 3 liters in volume in order to minimize volume changes when testing McLeod gages. The manometer shall be

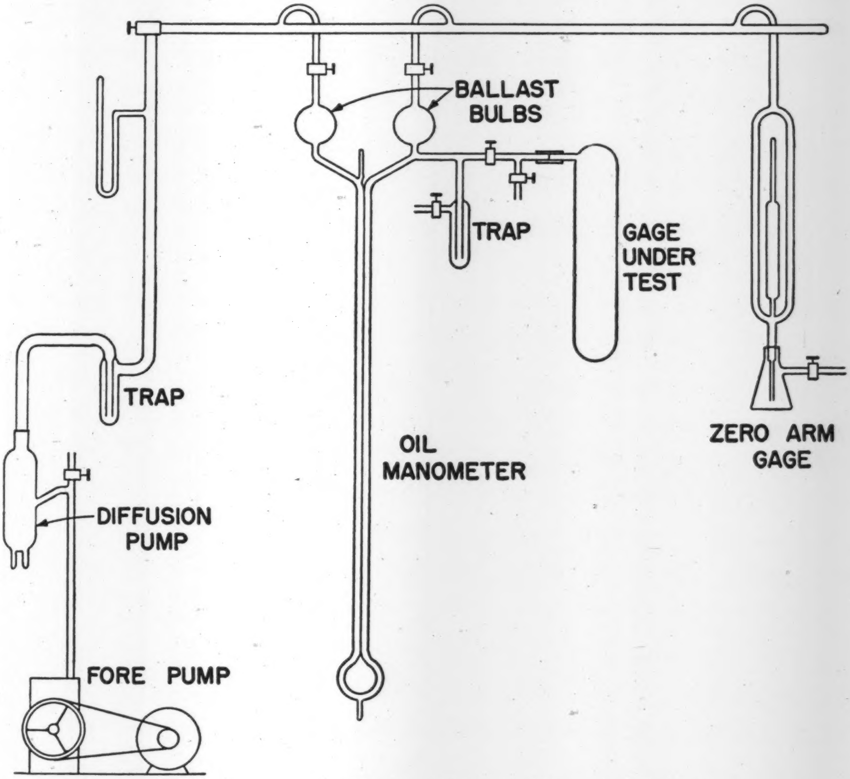

FIG. 1.—System for Checking Pressure Measuring Devices.

built of tubing having an inside diameter of not less than 10 mm. It shall be filled with a low density, low vapor pressure fluid such as butyl-phthalate or Apiezon "B" oil.[8] The density of the fluid at the temperature of use shall be accurately known. After filling, the manometer and fluid shall be thoroughly degassed by gentle heating under vacuum. In operation, the gage or device to be tested shall be attached to the right arm of the manometer through suitable connections. Evacuate the entire system and test for leaks. Close the stopcock at the top of the right arm of the manometer, but continue pumping on the opposite arm. Slowly admit air through a drying trap until the pressure on the right arm is at the point desired for testing. Allow about 5 min for drainage and stabilization of the manometers arms. Using a cathetometer, carefully read the difference in level of the manometer arms. Observe the reading of the gage being tested and check the pressure on the reference arm to see that it is less than 1 micron.

Calculate the pressure in millimeters of mercury by using the ratio of the densities of mercury and the manometric fluid at the temperature of test. If the manometer can be read with an accuracy of 0.5 mm, pressure changes of 0.03 mm of mercury can be detected with precision. For use in pressure ranges exceeding the practical length of an oil manometer, a mercury manometer may be added to the system and used in the same manner.

New Figure.—Add new Fig. 10 showing a system for checking pressure measuring devices.

REVISION OF TENTATIVES

Committee D-2 recommends revision of the following ten tentatives, as indicated:

D 910 - 50 T.[9] Specifications for Aviation Gasolines, revised as follows to include one new grade of aviation gasoline:

Section 2.—Change to read: "Four grades of aviation gasoline are provided, known as:

Grade 80–87
Grade 91–98
Grade 100–130
Grade 115–145."

Section 9.—Change Paragraph (*c*) to read: "(*c*) *Color.*—Color comparison may be made by any suitable apparatus or by visual examination using identical transparent containers for the fuel sample and for the appropriate Air Force-Navy Aeronautical color standards for maximum intensity and minimum intensity (Note 1). Samples of Air Force-Navy Aeronautical color standards which have been exposed to light for more than 24 hr shall not be used for this test."

Change the first sentence of NOTE 1 in Section 9 to read: "Standard maximum and minimum intensity Air Force-Navy Aeronautical fuel color samples are available in 1-gal metal containers and may be obtained upon request to the Commanding General, Air Materiel Command, Wright Field, Dayton, Ohio."

Table I.—Revise as shown in the accompanying Table I.

D 975 - 50 T.[9] Specifications for Diesel Fuel Oils, revised as follows:

Title.—Revise the title to read "Tentative Classification of Diesel Fuel Oils." Replace the word "specifications" by the word "classification" throughout the method.

Section 2.—Change the word "specified" to read "classified."

Table I.—Change the flash point specification for the No. 2-D grade diesel fuel from "100 F or legal" to "125 F or legal." Change Copper Strip Corrosion, max, of the No. 1-D and 2-D fuels from "Negative" to "No. 3" with a footnote reading 'a No. 4 ASTM Copper Strip or darker constitutes grounds for rejection of the fuel.'

Delete first sentence of footnote *a*.

Add a footnote *d* to read: "A method for estimating 'Calculated Cetane Index' is available in the 1951 Supplement to the ASTM Manual of Engine Test Methods for Rating Fuels."

D 611 - 47 T.[2] Test for Aniline Point and Mixed Aniline Point of Petroleum Products. The proposed revisions to this Method are so extensive that the revised method is appended hereto.[10] These changes are made to permit the determination of aniline point and mixed aniline point of different types of petroleum products.

D 721 - 49 T.[2] Test for Oil Content of Paraffin Wax, revised as follows:

Section 3 (*a*).— Change to read as follows: "*3* (*a*) ***Filter Stick and Assembly***, consisting of a 10-mm diameter medium-porosity sintered-glass filter stick, provided with an air

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[8] Available commercially from James G. Biddle Co., 1213 Arch St., Philadelphia, Pa.

[9] 1950 Supplement to Book of ASTM Standards, Part 5.

[10] The revised method was accepted by the Society and appears in the 1951 Supplement to Book of ASTM Standards, Part 4.

TABLE I.—DETAILED REQUIREMENTS[a] FOR AVIATION GASOLINES.

	Grade 80–87	Grade 91–98	Grade 100–130	Grade 115–145	Test Method[b]
Knock value, min, octane number, lean rating	80	91	100	*iso*octane plus 0.47 ml tetraethyllead per gallon	ASTM D 614
Knock value, min, octane number, rich rating	87	98	*iso*octane plus 1.28 ml of tetraethyllead per gallon	*iso*octane plus 2.8 ml of tetraethyllead per gallon	ASTM D 909
Color	red[c]	blue	green	purple	Section 9 (*c*)
Dye Content:					
Permissible blue dye,[d] max, mg per gal	0.75	8.5	7.0	7.0	
Permissible yellow dye,[e] max, mg per gal	none	none	7.0	none	
Permissible red dye,[f] max, mg per gal	8.65	none	none	3.27	
Tetraethyllead,[g] max, ml per gal	0.5[c]	2.0	3.0	4.6	ASTM D 526
Net heat of combustion,[h] min, Btu per lb	18 700	18 700	18 700	18 800	ASTM D 240

REQUIREMENTS FOR ALL GRADES

Distillation temperature, deg F:		
10 per cent evaporated, max	158	ASTM D 86
50 per cent evaporated, max	221	
90 per cent evaporated, min	212	
90 per cent evaporated, max	257	
Final boiling point, max, deg F	338	
Sum of 10 and 50 per cent evaporated temperatures, min, deg Fahr	307	
Distillation recovery, min, per cent	97	
Distillation residue, max, per cent	1.5	
Distillation loss, max, per cent	1.5	
Acidity of distillation residue	shall not be acid	ASTM D 1093
Vapor pressure, max, lb	7.0	ASTM D 323
Corrosion (Copper strip)	no worse than No. 1 comparison standard	ASTM D 130
Gum by copper dish, max, mg per 100 ml	5	Section 9 (*j*)
Potential gum (5 hr aging gum),[i] max, mg per 100 ml	6	ASTM D 873
Visible lead precipitate,[j] max, mg per 100 ml	3	ASTM D 873
Sulfur, max, per cent	0.05	ASTM D 90
Freezing point, max, deg F	minus 76	Section 9 (*m*)
Water tolerance	volume change not to exceed ± 2 ml	ASTM D 1094
Permissible gum inhibitors,[k] max, lb per 1000 bbl (42 gal)	4.2	

[a] Requirements contained herein are absolute and are not subject to correction for tolerance of the test methods. If multiple determinations are made, average results shall be used.

[b] The test methods indicated in this table are described or referred to in Section 9.

[c] If mutually agreed upon between the vendor and the purchaser, Grade 80–87 may be required to be free from tetraethyllead. In such a case, the fuel shall not contain any dye and the color as determined in accordance with Standard Method of Test for Color of Refined Petroleum Oil by Means of Saybolt Chromometer (ASTM Designation: D 156) shall not be darker than plus 20.

[d] The only blue dye which shall be present in the finished fuel shall consist essentially of an alkyl-substituted anthraquinone.

[e] The only yellow dye which shall be present in the finished fuel shall be Yellow-N, *p*-dimethylaminoazobenzene (National Color Index Reference No. 19).

[f] The only red dye which shall be present in the finished fuel shall be Red-0, 2, 3′-dimethylazobenzene-4′-azo-B-naphthol (Color Index No. 258).

[g] The tetraethyllead shall be added in the form of an antiknock mixture containing not less than 61 per cent by weight of tetraethyllead and sufficient ethylene dibromide to provide two bromine atoms per atom of lead. The balance shall contain no added ingredients other than kerosine, and approved inhibitor, and blue dye, as specified, herein.

[h] The net heat of combustion determination may be waived if the product of the gravity of the fuel as determined in accordance with the Standard Method of Test for Gravity of Petroleum and Petroleum Products by Means of the Hydrometer (ASTM Designation: D 287) in degrees API and the aniline point of the fuel as determined in accordance with Tentative Method of Test for Aniline Point and Mixed Aniline Point of Petroleum Products (ASTM Designation: D 611) in degrees Fahrenheit is equal to or above 7400 in the case of Grades 80–87, 91–98, and 100–130 and equal to or above 8200 in the case of Grade 115–145. The product of the aniline point and the API gravity shall be known as the aniline-gravity constant.

[i] If mutually agreed upon between the vendor and the purchaser, aviation gasoline may be required to meet a 16-hr aging gum test (ASTM Method D 873) instead of the 5-hr aging gum test. In such a case, the gum content shall not exceed 10 mg per 100 ml and the visible lead precipitate shall not exceed 4 mg per 100 ml. In such fuel the permissible gum inhibitors shall not exceed 8.4 lb per 1000 bbl (42 gal).

[j] The visible lead precipitate requirement applies only to leaded fuels.

[k] Permissible gum inhibitors are as follows:

N, N′ di-secondary-butyl-para-phenylenediamine
2, 4, dimethyl-6-tertiary-butylphenol
2, 6-ditertiary butyl, 4-methylphenol.

These inhibitors may be added to the gasoline separately or in combination, in total concentration not to exceed 4.2 lb of inhibitor (not including weight of solvent) per 1000 bbl (42 gal).

pressure inlet tube and delivery nozzle. It is provided with a ground-glass joint to fit a 25 by 170-mm test tube. The dimensions for a suitable filtration assembly are shown in Fig. 1." Retain Note 2 without change.

Section 3 (*f*).—Change to read as follows:

"(*f*) *Thermometers.*—Oil in Wax Thermometers having the range of −35 to +70 F.[11]

Section 3 (*i*).—Add new Section 3 (*i*), to read as follows:

"(*i*) *Analytical Balance*, capable of reproducing weights to 0.1 mg. The sensitivity should be adjusted so that 0.1 mg will deflect the pointer one-half division on the pointer scale."

Section 6 (*b*).—Revise the first sentence to read as follows: "Pipet 15 ml of methylethylketone into the test tube and place the latter just up to the level of its contents in a hot-water or steam bath."

Section 6 (*c*).—In the first sentence, change "−30 to −35 F" to read "−30 ± 2 F".

Section 6 (*d*).—In the first sentence, change "−30 to −35 F" to read "−30 ± 2 F". Revise the second sentence to read "Seat the ground-glass joint of the filter so as to make an air-tight seal."

Figure 1.—Change Fig. 1 to illustrate the ground-glass stoppered assembly, rather than the cork-stoppered assembly now shown.

D 808 - 50 T.[9] Test for Chlorine in Lubricating Oils and Greases by the Bomb Method. The proposed revisions to this method are so extensive that the revised method is appended hereto.[12]

D 936 - 49 T.[2] Test for Aromatic Hydrocarbons in Olefin-Free Gasolines by Silica Gel Adsorption. The proposed revisions to this method are so extensive that the revised method is appended hereto.[12]

D 972 - 48 T.[2] Test for Evaporation Loss of Lubricating Greases and Oils revised as appended hereto.[12]

D 974 - 48 T.[2] Test for Neutralization Value (Acid and Base Numbers) by Color-Indicator Titration, revised as follows:

Section 1 (*a*).—Add the following note:

"NOTE 2.—This method is not suitable for measuring the basic constituents of many basic additive-type lubricating oils. ASTM Method D 664, Test for Neutralization Value (Acid and Base Numbers by Electrometric Titration), can be used for this purpose." Renumber subsequent notes accordingly.

Section 7 (*b*).—Change the last sentence to read as follows: "Consider the end point definite if the color change (Note 5) persists for 15 sec or if it reverses with two drops of $0.1\ N$ HCl."

D 1016 - 50 T[9]. Test for Determination of Purity from Freezing Points. The material proposed for incorporation in this method consists of the information necessary for the determination of purity of butane, isobutane and isobutene, as appended hereto.[12]

D 1088 - 50 T[9]. Test for Boiling Point Range of Polymerization-Grade Butadiene, revised as follows:

Section 3 (*b*).—Change to read:

(*b*) *Thermometer.*[13]—A thermometer which meets the following specifications:

Liquid	mercury
Filling above Liquid	nitrogen gas
Temperature Range	−10 to +10 C
Subdivisions	0.1 C
Total Length	160 ± 3 mm
Stem Diameter	6.0 to 6.5 mm
Bulb Diameter	5.5 mm min to not greater than stem diameter
Bulb Length	9 to 13 mm, max
Bottom of Bulb to Graduation	
Line at	−10 C
Distance	28 to 36 mm
Top of Thermometer to Graduation	
Line at	+10 C
Distance	35 to 51 mm
Expansion Chamber	long narrow type to permit heating to 100 C. Length of unchanged capillary between highest graduation and base of expansion chamber shall be not less than 10 mm.
Top Finish	plain
Graduation Lines at Each	0.5 C
Graduations Numbered at Each Multiple of	1.0 C
Immersion	total
Scale Error at any Point when Standardized Shall not Exceed	0.1 C

REVISION OF STANDARDS AND REVERSION TO TENTATIVE

Committee D-2 recommends that the following three standards be revised as appended hereto[14] and reverted to tentative:

D 447 - 41. Test for Distillation of Plant Spray Oils,

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[11] See the Proposed Specifications for Oil in Wax Thermometer, as an appendix to the Tentative Method of Test for Oil Content of Paraffin Wax (D 721 - 51 T) which appears in the 1951 Supplement to Book of ASTM Standards, Part 5.

[12] This revised method was accepted by the Society and appears in the 1951 Supplement to Book of ASTM Standards, Part 5.

[13] This thermometer is available from the H-B Instrument Co., Philadelphia, Pa.

[14] These revised methods were accepted by the Society and appear in the 1951 Supplement to Book of ASTM Standards, Part 5.

D 483 – 40. Test for Unsulfonated Residue of Plant Spray Oils, and

D 524 – 42. Test for Carbon Residue of Petroleum Products (Ramsbottom Carbon Residue). This Method has been extensively revised.

REVERSION OF STANDARD TO TENTATIVE

Committee D-2 recommends that Standard Method D 157 – 42, Test for Steam Emulsion of Lubricating Oils, be reverted to the tentative status, as the method is believed to be obsolete and in need of revision.

ADOPTION OF TENTATIVES AS STANDARD

Committee D-2 finds that the following tentatives represent the best present-day practices, in each case, and recommends that they be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Methods of Test for:

D 874 – 47 T. Sulfated Residue from New Lubricating Oils,

D 908 – 49 T. Knock Characteristics of Motor Fuels by the Research Method,

D 1017 – 49 T. Benzene and Toluene by Ultraviolet Spectrophotometry,

D 1019 – 49 T. Total Olefinic Plus Aromatic Hydrocarbons in Petroleum Distillates, and

D 1092 – 50 T. Apparent Viscosity of Lubricating Greases.

The committee also recommends that Method D 1026 – 49 T, Test for Sodium in Lubricating Oils and Lubricating Oil Additives, be adopted as standard with the following revisions:

Section 2.—Revise the first sentence of Section 2 by omission of the words in brackets and the addition of the italicized words to read as follows: "The sample is [completely] oxidized by dry ashing; the oil is allowed to burn freely until only coke or tar remains, and the free carbon on the sides of the dish is ignited at a dull red heat (550 C) *for 60 min.* [until all carbonaceous matter has disappeared.]"

Section 5 (*b*).—Revise the last sentence to read as follows by omission of the words in brackets and the addition of the italicized words: "Place the dish in a cool furnace (below 300 C), gradually increase the temperature to 550 ± 50 C, and continue the ignition [until the oxidation of carbon is complete] *at this temperature for 60 min.*"

NOTE 3.—Add new Note 3 at the end of Section 5 (*b*) to read as follows; renumbering the subsequent notes accordingly:

"NOTE 3.—Small amounts of carbonaceous material remaining after ignition do not interfere in the method."

REVISION OF STANDARDS, IMMEDIATE ADOPTION

Committee D-2 recommends for immediate adoption revisions in the following three standards and accordingly asks for a nine-tenths affirmative vote at the Annual Meeting in order that these revisions may be referred to letter ballot of the Society:

D 56 – 36,[2] Test for Flash Point by Means of the Tag Closed Tester. This method has been revised extensively to make it suitable for testing present-day products and to take advantage of the latest developments in test apparatus. The revised method is appended to this report.[12]

D 129 – 50,[9] Test for Sulfur in Petroleum Products and Lubricants by the Bomb Method.

Section 1.—Change the scope to read as follows: "1. *Scope.*—This method describes a procedure for the determination of sulfur in petroleum products, including lubricating oils containing additives, additive concentrates, and lubricating greases that cannot be burned completely in a wick lamp. The method is applicable to any petroleum product sufficiently low in volatility that it can be weighed accurately in an open sample boat."

Section 5 (*a*).—Change Note 1 by replacing the reference to 00 Hupert's paper with a reference to Grit No. 2/0 paper. Change Footnote 3 to read: "Grit No. 2/0 paper may be purchased from the Behr-Manning Co, Troy, N. Y. Chromic oxide may be purchased from the J. T. Baker Co, Phillipsburg, N. J."

D 288 – 49,[2] Definitions of Terms Relating to Petroleum. Include the following definitions:

Asphalt.—Black to dark brown solid or semi-solid cementitious material, which gradually liquefies when heated, in which the predominating constituents are bitumens, all of which occur in the solid or semi-solid form in nature or are obtained by refining petroleum or which are combinations of the bitumens mentioned with each other or with petroleum or derivatives thereof.

Native Asphalt.—Asphalt occurring as such in nature.

Petroleum Tar.—A viscous black or dark-brown product obtained in petroleum refining which will yield a substantial quantity of solid residue when partially evaporated or fractionally distilled. The term "tar" should never be applied to materials derived from crude petroleum or fractions thereof without the prefix "petroleum."

Tar.—Delete. This term should not be applied to petroleum products.

Petroleum Spirits.—Delete the second sentence of the Note under this definition.

The committee also recommends that Method D 664 - 49,[2] Test for Neutralization Value (Acid and Base Numbers) by Electrometric Titration be revised by the addition of the following new section and the subsequent sections be relettered accordingly:

8. *Preparation of Used Oil Samples.*—(*a*) Strict observance of the sampling procedure described in Paragraph (*b*) is necessary, since the sediment itself is acidic or basic or has adsorbed acidic or basic material from the sample. Failure to obtain a representative sample causes serious errors.

(*b*) Heat the sample of used oil to 60 ± 5 C in the original container and agitate until all sediment is homogeneously suspended in the oil (Note 5). If the original container is of opaque material, or if it is more than three-fourths full, transfer the entire sample to a clear glass bottle having a capacity at least one-third greater than the volume of the sample, and transfer all traces of sediment from the original container to the bottle by violent agitation of portions of the sample in the original container. After complete suspension of all sediment, strain the sample or a convenient aliquot through a 100-mesh screen for the removal of large contaminating particles.

NOTE 4.—As used oil may change appreciably in storage, samples should be tested as soon as possible after removal from the lubricating system and the dates of sampling and testing should be noted.

EDITORIAL REVISIONS

Committee D-2 recommends the following editorial revisions in the two methods:

D 128 - 47. Methods of Analysis of Grease, revision of Section 1 on Scope to read as follows:

1. *Scope.*—These methods of analysis are suitable for greases of the conventional type comprised essentially of petroleum oil and soap. The constituents which can be determined are soap, unsaponifiable matter (petroleum oil, etc.), water, free alkalinity, free fatty acid, fat, glycerine and filler.

NOTE.—The methods described herein are best used by an experienced grease analyst.

These methods are not suitable for greases made with non-soap thickeners, with oily constituents other than petroleum oil or with hydroxy fatty acid soaps. The experienced analyst, however, may be able to adapt these techniques or portions thereof to the analysis of such greases.

D 942 - 50. Test for Oxidation Stability of Lubricating Greases by the Oxygen Bomb Method, changing Section 6 (*d*) on Procedure to read as follows:

(*d*) Start timing at the moment of immersion of the bomb in the oil bath, and continue the oxidation for the time period specified.

TENTATIVES CONTINUED WITHOUT REVISION

Committee D-2 has reviewed the following 22 tentatives, which have stood for two or more years without revision, and, for good and sufficient reasons, discussed within the committee, recommends their continuation without change:

Tentative Specifications for:

D 396 - 48 T. Fuel Oils.

Tentative Methods of:

D 94 - 48 T. Test for Saponification Number of Petroleum Products by Color-Indicator Titration,

D 155 - 45 T. Test for Color of Lubricating Oil and Petrolatum by Means of ASTM Union Colorimeter,

D 445 - 46 T. Test for Kinematic Viscosity,

D 526 - 48 T. Test for Tetraethyllead in Gasoline,

D 613 – 48 T. Test for Ignition Quality of Diesel Fuels by the Cetane Method,
D 614 – 49 T. Test for Knock Characteristics of Aviation Fuels by the Aviation Method,
D 855 – 46 T. Analysis of Petroleum Sulfonates,
D 875 – 46 T. Test for Olefins and Aromatics in Petroleum Distillates,
D 892 – 46 T. Test for Foaming Characteristics of Crankcase Oils,
D 909 – 48 T. Test for Knock Characteristics of Aviation Fuels by the Supercharge Method,
D 937 – 49 T. Test for Penetration of Petrolatum,
D 943 – 47 T. Test for Oxidation Characteristics of Inhibited Steam-Turbine Oils,
D 976 – 48 T. Test for Color of US Army Motor Fuel (All-Purpose) by Means of an ASTM Color Standard,
D 1015 – 49 T. Test for Measurement of Freezing Points for Evaluation of Purity,
D 1018 – 49 T. Test for Hydrogen in Petroleum Fractions by the Lamp Method,
D 1020 – 49 T. Test for Acetylene in Polymerization-Grade Butadiene by Silver Nitrate Method,
D 1021 – 49 T. Test for Oxygen in Butadiene Vapors by Manganous Hydroxide Method,
D 1022 – 49 T. Test for Peroxides in Butadiene by Ferrous-Titanous Method,
D 1023 – 49 T. Test for Separation of Residue from Butadiene,
D 1024 – 49 T. Test for Butadiene Dimer in Polymerization-Grade Butadiene, and
D 1025 – 49 T. Test for Nonvolatile Residue of Polymerization-Grade Butadiene.

AMERICAN STANDARDS

Committee D-2 recommends that all new ASTM standards which are recommended in this report and which may be accepted by the Society be referred to ASA Sectional Committee Z-11 on Petroleum Products and Lubricants for consideration for approval as American Standard. In addition, Committee D-2 recommends that the following methods which have been reverted to tentative be considered for reapproval as American Standards: D 524, Method of Test for Carbon Residue of Petroleum Products (Ramsbottom Carbon Residue); D 447, Method of Test for Distillation of Plant Spray Oils; and D 483, Method of Test for Unsulfonated Residue of Plant Spray Oils.

VISCOSITY AT HIGH RATES OF SHEAR

A Symposium in Methods of Measuring Viscosily at High Rates of Shear was held during the year and the papers presented have been published as Special Technical Publication No. 111.

The recommendations appearing in this report have been submitted to letter ballot vote of the committee, the results of which will be reported at the Annual Meeting.

This report has been submitted to letter ballot of the committee, which consists of 106 members; 76 members returned their ballots, of whom 70 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

O. L. MAAG,
Chairman.

W. T. GUNN,
Secretary.

APPENDIX I

PROPOSED METHOD OF TEST FOR PENETRATION OF LUBRICATING GREASES WORKED MORE THAN 60 STROKES[1]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

The following method is a proposed addition to the Appendix in the Standard Method of Test for Cone Penetration of Lubricating Grease (D 217 - 48):[2]

Procedure

A4. To obtain information on the penetrations of greases after being worked for more than 60 strokes, proceed as follows:

(*a*) Fill the cup and adjust to 77 ± 1 F, as directed in Section 5.[3] Work for 60 strokes and, if desired, determine the penetration as directed in Section 5 (*d*).

(*b*) Using the same cup of grease, perform the desired additional working, observing the following deviations:

(*1*) Operate the worker mechanically.

(*2*) Perform the working in a constant-temperature room or air chamber maintained at 77 ± 1 F.

(*3*) Upon completing the additional working, open the petcock and determine the temperature of the grease with the dial thermometer.

(*4*) Prepare the sample, and immediately determine the penetration, as directed in Section 5 (*c*) and (*d*).

(*c*) Place the cup and contents in a water bath maintained at 77 ± 1 F, with the water level coinciding with the lower limit of the threaded portion of the cup. At each 5-min interval thereafter, proceed as follows:

(*1*) Scoop out with the spatula a generous portion of the grease and return it, inverted, to the cup, so that portions of the grease from the bottom will be brought to the surface, and the portions previously at the surface will be buried in the cup. Note the temperature of the grease at each 5-min period and, as soon as the temperature has attained 77 ± 1 F, proceed as directed in Item (*2*).

(*2*) Immediately work the sample of grease for 60 strokes and determine the penetration as directed above, beginning with the fifth sentence of Section 5 (*b*), and continuing through Section 5 (*d*).

(*d*) Report the following for each degree of working:

(*1*) Total number of strokes,

(*2*) Temperature immediately after working,

(*3*) Penetration immediately after working, and

(*4*) Penetration after cooling to 77 F and working 60 additional strokes.

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[1] This proposed method is under the jurisdiction of the ASTM Committee D-2 on Petroleum Products and Lubricants. Published as information June, 1951.

[2] 1949 Book of ASTM Standards, Part 5.

[3] This and subsequent section numbers refer to the Standard Method D 217 - 48, see 1949 Book of ASTM Standards Part 5, p. 816.

APPENDIX II

PROPOSED METHOD OF TEST FOR DENSITY AND SPECIFIC GRAVITY OF LIQUIDS TO THE FIFTH PLACE BY THE BINGHAM PYCNOMETER[1]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race Street, Philadelphia 3, Pa.

Scope

1. This method describes a test procedure for the measurement of density or specific gravity of any material that can be handled in a normal manner as a liquid at the desired test temperature. Its application is restricted to liquids having vapor pressures less than 600 mm of mercury and having viscosities less than 30 centistokes at 100 F. The method is designed to provide values having an accuracy of 0.00003 g per ml at any desired temperature between 0 and 95 C.

Definitions

2. (*a*) *Density* is the weight in vacuo, (that is, the mass) of a unit volume of the material at any given temperature. In this method the unit of mass is the gram, and the unit of volume, the milliliter; unless otherwise specified, the temperature scale used is the Centigrade.

(*b*) *Specific Gravity* is the ratio of the mass (weight in vacuo) of a given volume of material at a temperature, t_1, to the mass of an equal volume of water at a reference temperature, t_2; or it is the ratio of the density of the material at t_1 to the density of water at t_2. When the reference temperature is 4.00 C, the temperature at which the relative density of water is unity, specific gravity and density are numerically equal.

Outline of Method

3. The liquid sample is introduced into a pycnometer, equilibrated to the desired temperature, and weighed. The specific gravity or density is then calculated from this weight and the previously determined weight of water that is required to fill the pycnometer at the same temperature, both weights being corrected for the buoyancy of air so that the resulting value represents a ratio of masses.

Apparatus

4. (*a*) *Pycnometer*, Bingham-type, conforming to the dimensions given in Fig. 1, constructed of Pyrex glass and having a total weight not exceeding 30 g.

(*b*) *Constant Temperature Bath*, provided with suitable pycnometer holders or clips and means for maintaining temperatures constant to ±0.01 C in the desired range.

(*c*) *Bath Thermometer*, graduated in 0.1-C subdivisions and standardized for the ice point and the range of use to the nearest 0.01 C. ASTM Kinematic Viscosity Thermometers 44F and 45F as prescribed in ASTM Specifications E 1,[2] designed for tests at 68 F and 77 F, are recommended. A standardized platinum resistance thermometer may also be used, and offers the best means for observing minute temperature changes in the bath. Whichever means are available, it must be realized that for most hydrocarbons the density coefficient is about 0.0008 units per deg Cent, and therefore an error of ±0.013 C would cause an error of ±0.00001 in density.

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[1] This proposed method is under the jurisdiction of the ASTM Committee D-2 on Petroleum Products and Lubricants. Published as information, June, 1951.

[2] 1951 Supplement to Book of ASTM Standards, Part 5.

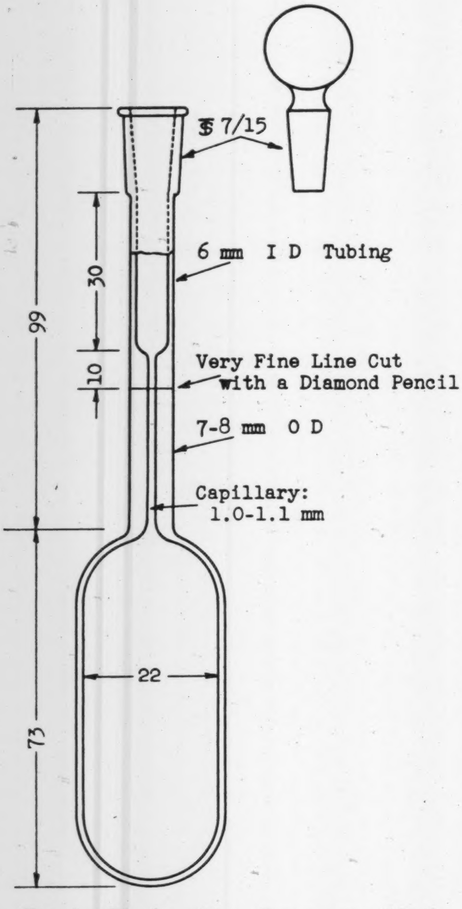

FIG. 1.—Bingham-Type Pycnometer, 25 ml.

(*d*) *Hypodermic Syringe,* 30-ml capacity, of chemically-resistant glass, equipped with a 6-in. needle made of stainless steel tubing as shown in Fig. 2.

(*e*) *Draw-Off Needle* made of stainless steel tubing as shown in Fig. 2.

(*f*) *Solvent-Cleaning Assembly* as shown in Fig. 3.

(*g*) *Chromic Acid Cleaning Apparatus* similar to that shown in Fig. 4.

(*h*) *Balance,* capable of reproducing weighings within 0.1 mg, and having a sensitivity which causes the pointer to be deflected 2 or 3 scale divisions per 1 mg when carrying a load of 30 g or less on each pan. The balance should be located in a room shielded from drafts and fumes and in which the temperature changes between related weighings (empty and filled pycnometer) do not cause a significant change in the ratio of the balance arms. Otherwise weighings shall be made by the method of substitution, in which the calibrated weights and pycnometer are alternately weighed on the same balance pan. The same balance shall be used for all related weighings.

(*i*) *Weights,* whose relative values are known to the nearest 0.05 mg or better. The same set of weights shall be used for the calibration of the pycnometer and the determination of densities.

Preparation of Apparatus

5. (*a*) Thoroughly clean the pycnometer with hot chromic acid cleaning solution by means of the assembly shown in Fig. 4. Mount the apparatus firmly and connect the trap to the vacuum. Warm the necessary amount of cleaning acid in the beaker, place the pycnometer on the ground joint, and evacuate by opening the stopcock to vacuum. Fill the pycnometer with acid by turning the stopcock, repeat several times or remove the filled pycnometer, and allow it to stand for several hours at 50 to 60 C. Remove the acid from the pycnometer by evacuation, empty the acid from the trap, and flush the pycnometer with water. Cleaning should be made in this manner whenever the pycnometer is to be calibrated or whenever liquid fails to drain cleanly from the walls of the pycnometer or its capillary. Ordinarily, the pycnometer may be cleaned between determinations by washing with a suit-

able solvent, rinsing with pure, dry acetone, followed by isopentane, and vacuum drying.

(*b*) Transfer the pycnometer to the cleaner assembly shown in Fig. 3, with vacuum line and trap attached to the side tube as indicated. Place the pycnometer on the cleaner with the upper hypodermic needle extending upward into the pycnometer, and press the edge of the ground joint on the rubber stopper until the vacuum holds it in place. Draw out all the liquid or sample. Immerse the lower end of the hypodermic tube in a suitable solvent and draw 20 to 25 ml through the pycnometer. Leaving the pycnometer in place, draw air through it until it is dry. Clean the hypodermic syringe with the same apparatus.

Calibration of Pycnometer

6. Proceeding as directed in Section 7, determine the weight of freshly-boiled and cooled distilled water (distilled from alkaline permanganate through a tin condenser) held by the pycnometer when equilibrated to volume at the bath temperature to be used in the determination. Repeat until at least three values agree to ±0.2 mg.

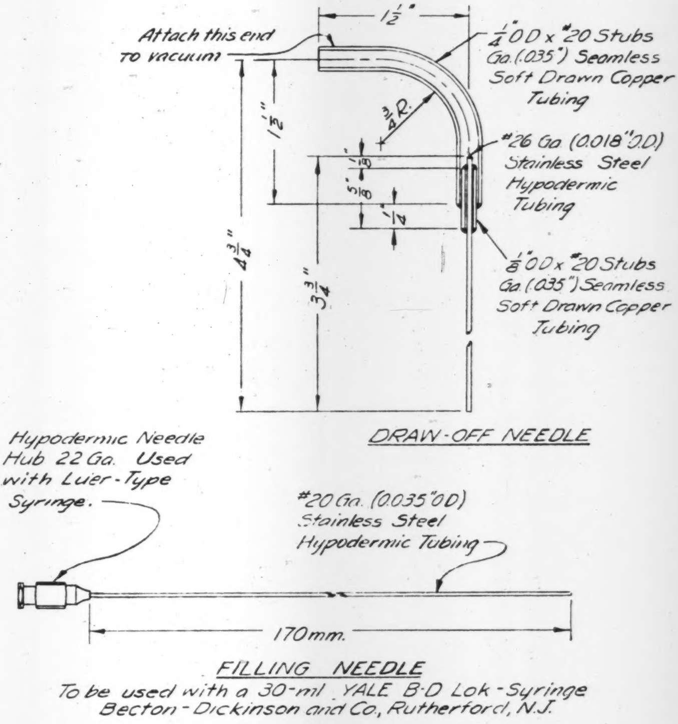
Attach this end
To vacuum
1 1/2"
1/4" OD x #20 Stubs Ga.(.035") Seamless Soft Drawn Copper Tubing
3/4 R.
1 1/2"
#26 Ga (0.018" OD) Stainless Steel Hypodermic Tubing
5/8"
1/8"
1/4"
4 3/4"
3 3/4"
1/8" OD x #20 Stubs Ga (.035") Seamless Soft Drawn Copper Tubing
DRAW-OFF NEEDLE
Hypodermic Needle Hub 22 Ga. Used with Luer-Type Syringe.
#20 Ga. (0.035" OD) Stainless Steel Hypodermic Tubing
170 mm.
FILLING NEEDLE
To be used with a 30-ml YALE B-D Lok-Syringe
Becton-Dickinson and Co, Rutherford, N.J.

FIG. 2.—Accessories for Bingham-Type Pycnometer.

Procedure

7. (*a*) Using another 25-ml pycnometer as a tare (Note 1), weigh the clean, dry pycnometer to 0.1 mg and record the weight.

NOTE 1.—It is convenient to use the lightest of a set of pycnometers as a tare. For best results the treatment and environment of both pycnometer and tare should be identical for some time prior to weighing.

(*b*) Cool the sample to 5 to 10 C below the test temperature, and fill the clean 30-ml hypodermic syringe. Transfer the sample to the pycnometer through the filling needle; avoid trapping air bubbles (Note 2) in the bulb or capillary of the pycnometer. If any are present, draw them into the syringe where possible. Also remove with the syringe or draw-off needle any liquid above the calibration mark in the capillary or overflow reservoir. Dry the remainder with a cotton fiber pipe cleaner or cotton swab which has been dampened slightly with acetone.

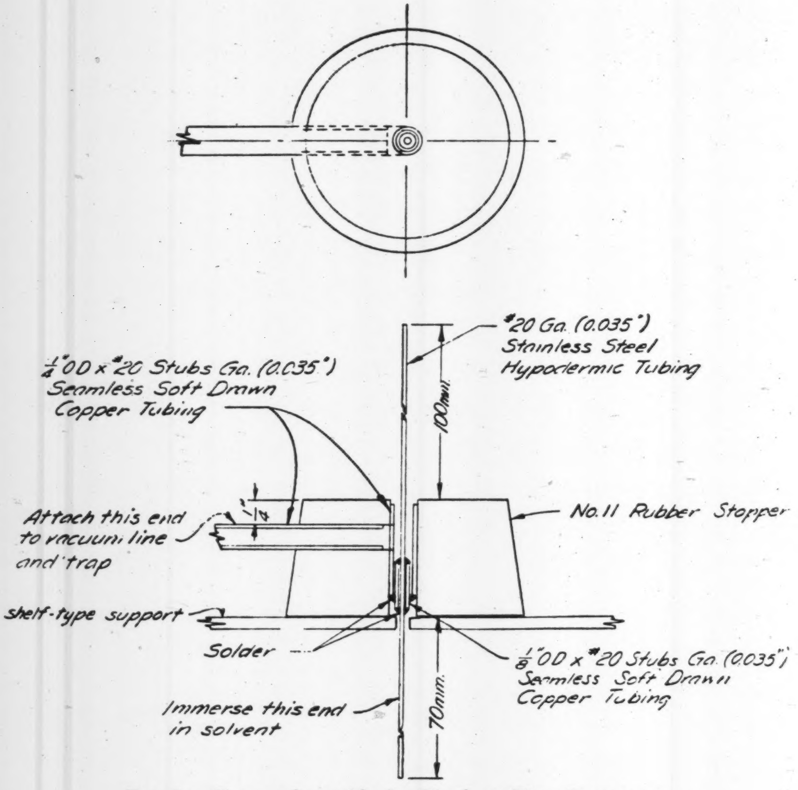
1/4" OD x #20 Stubs Ga. (0.035") Seamless Soft Drawn Copper Tubing
#20 Ga. (0.035") Stainless Steel Hypodermic Tubing
100 mm.
1/4
Attach this end to vacuum line and trap
No. 11 Rubber Stopper
shelf-type support
Solder
1/8" OD x #20 Stubs Ga. (0.035") Seamless Soft Drawn Copper Tubing
Immerse this end in solvent
70 mm.

FIG. 3.—Cleaner Assembly for Bingham-Type Pycnometer.

NOTE 2.—For work of highest accuracy on pure compounds, dissolved air may be removed from the sample by repeated freezing and remelting of the sample under vacuum in the pycnometer.

(*c*) Close the pycnometer with the glass stopper and immerse it to a point above the calibration mark in the constant-temperature bath adjusted to a constancy of ±0.01 C at the desired temperature. Periodically, or before the liquid expands into the overflow chamber, remove the stopper, raise the pycnometer sufficiently to expose the calibration mark to view, and readjust the liquid level to the mark by withdrawing liquid through the steel draw-off needle until expansion has stopped, indicating that the liquid has reached the temperature of the thermostat. Do not allow the liquid to expand more than 1 cm above the calibration mark at any time, to minimize errors caused by faulty drainage. Allow the contents to equilibrate an additional 10 min and draw the level down exactly to the calibration line, avoiding parallax and using a magnifier, if necessary, to obtain good visibility. Remove any liquid adhering to the walls above the calibration mark, with the draw-off needle or pipe cleaner, depending upon the volatility of the sample. Portions in the overflow bulb may be removed with a cotton swab moistened with acetone.

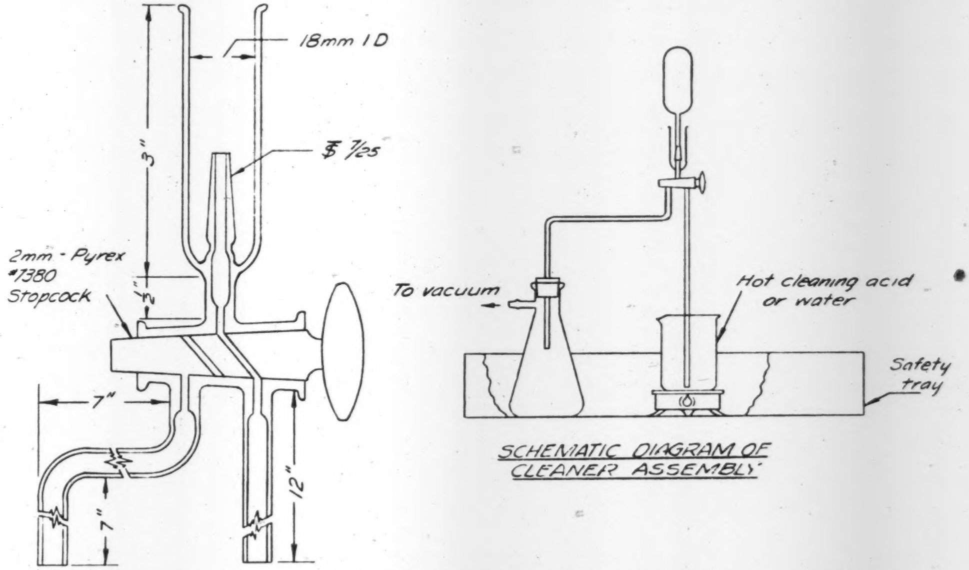

FIG. 4.—All-Glass Pycnometer Cleaner Assembly for Use with Hot Chromic Acid Cleaning Solution.

(*d*) Replace the glass stopper, remove the pycnometer from the bath, wash the outside surface with acetone, and dry thoroughly with a chemically clean, lint-free, slightly damp cloth. Place the pycnometer in or near the balance case for 20 min and weigh to the nearest 0.1 mg.

NOTE 3.—In atmospheres of low humidity (60 per cent or lower), drying the pycnometer by rubbing with a dry cotton cloth will induce static charges equivalent to a loss of about 1 mg in the weight of the pycnometer. This charge may not be completely dissipated in less than 30 min. The use of about 0.1-mg radium bromide- or polonium-coated foil in the balance case, or maintaining the relative humidity at 60 per cent or higher, aids in reducing weighing difficulties due to static charges.

(*e*) Note and record temperature of the balance, barometric pressure, and relative humidity.

Calculation

8. (*a*) Calculate the true density of the sample as follows:

Density, g per ml at deg Cent

$$= \frac{W_s\left(1 + \frac{d_a}{d_s} - \frac{d_a}{d_{wt}}\right)d_w}{W_w\left(1 + \frac{d_a}{d_w} - \frac{d_a}{d_{wt}}\right)}$$

where:

W_s = weight in air, in grams, of sample contained in the pycnometer at the test temperature,

W_w = weight in air, in grams, of the water contained in the pycnometer at the calibration temperature,

d_w = relative density of water at the calibration temperature, as obtained from Table I,
d_a = density of air in balance case at the time of weighing, as calculated from Section 8 (*c*),
d_{wt} = density of weights used in weighing the sample and water (brass = 8.4 g per ml, stainless steel = 7.75 g per ml), and
d_s = approximate density of sample or

$$\frac{W_s \times d_w}{W_w}$$

NOTE 4.—The formula assumes that the weighings of the pycnometer empty and filled are made in such a short time interval that the air density has not changed. If significant change should occur, the calçulated apparent weight of the sample, W_s, in this formula, must be corrected for the difference in air buoyancy exerted on the pycnometer as follows:

$$W_s = W^2{}_{PS} - \frac{W'p\left(1 + \frac{d_a'}{2.2} - \frac{d_a'}{d_{wt}}\right)}{\left(1 + \frac{d_a{}^2}{2.2} - \frac{d_a{}^2}{d_{wt}}\right)}$$

where:

$W^2{}_{PS}$ = weight of pycnometer and contained sample under second or final air density,
W_p' = weight of pycnometer in air of first density,
d_a' = density of air when weighing empty pycnometer,
d_a^2 = density of air when weighing filled pycnometer, and
d_{wt} and 2.2 = density of weights and Pyrex glass, respectively.

Likewise, if the pycnometer, empty and filled with water for calibration, is weighed under different air densities a similar correction for different air buoyancies shall be applied.

TABLE I.—RELATIVE DENSITY OF WATER

Temperature, deg Cent	Relative Density, g per ml	Temperature, deg Cent	Relative Density, g per ml	Temperature, deg Cent	Relative Density, g per ml
0	0.99987	21	0.99802	40	0.99224
3	999	22	0.99780	45	025
4	1.00000	23	756	50	0.98807
5	0.99999	24	732	55	573
10	973	25	707	60	324
15	913	26	0.99681	65	059
15.56	904	27	654	70	0.97781
16	0.99897	28	626	75	489
17	880	29	0.99597	80	183
18	862	30	567	85	0.96865
19	843	35	0.99406	90	534
20	823	37.78	0.99307	100	0.95838

(*b*) Calculate the specific gravity of the sample by dividing the density as obtained in Paragraph (*a*) by the relative density of water at the reference temperature obtained from Table I.

(*c*) Calculate the density of air in the balance room as follows:

Air density (d_a), g per ml,

$$= \frac{(B - 0.3783\,Hp)(0.000465)}{273 + t}$$

where:

B = barometric pressure, millimeters of mercury, corrected to 0 C,
H = relative humidity, decimal fraction,
p = vapor pressure of water at temperature t, in millimeters of mercury, and
t = room temperature, in degrees Centigrade.

NOTE 5.—If this method is to be used frequently, a considerable amount of calculation can be avoided by use of a gas density balance to determine the air density. Weigh a sealed 250-ml glass bulb at several different air densities and plot the weight against the air density. To determine the air density at some later time, weigh the bulb and read the air density from the point on the curve corresponding to the weight.

(*d*) To calculate the density or specific gravity at any test temperature, t, other than the calibration temperature, t_c (to correct for the cubical coefficient of thermal expansion of Pyrex glass), divide the value obtained in Paragraph (*a*) or (*b*) by the following expression:

$$1 + 9.6 \times 10^{-6}\,(t - t_c)$$

Report

9. In reporting density, give the test temperature and the units (for example, density, 20 C = x.xxxxx g per ml). In reporting specific gravity, give both the test temperature and the reference temperature, but no units (for example, specific gravity, 20/4 C = x.xxxxx). Carry all calculations to one digit beyond the last significant figure, but report the final result to the fifth decimal place (0.00001).

Precision and Accuracy

10. (*a*) Results, using the 25-ml Bingham-type pycnometer, should not differ from the mean by more than the following amounts:

Repeatability	**Reproducibility**
One Operator and Apparatus	Different Operators and Apparatus
0.00002	0.00003

(*b*) The accuracy of results shall not differ from the theoretical value by more than 0.00003.

APPENDIX III

PROPOSED METHOD OF TEST FOR SAPONIFICATION NUMBER OF PETROLEUM PRODUCTS (PARA-XYLENOL BLUE COLOR-INDICATOR METHOD)[1]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race Street, Philadelphia 3, Pa.

Scope

1. (*a*) This method of test is intended to determine the acidic and basic constituents produced upon saponification of petroleum products under the conditions of the test, as well as those contained in them prior to saponification.

(*b*) The method is applicable to materials that are soluble or nearly soluble in mixtures of benzene and isopropyl alcohol. It is intended particularly for the determination of the saponification number of new and used oils, electrical oils, and greases whether compounded or not. It is useful for the identification of unmixed animal or vegetable oils, and for the determination of the amount of fatty material in compounded products, provided the sample contains only one saponifiable fat or fatty oil and does not contain appreciable amounts of certain nonfatty compounds, such as those of sulfur, phosphorous, the halogens, nitrogen, and metals other than potassium or sodium, which consume alkali or acid under the conditions of the test.

(*c*) The method is not applicable to highly colored oils or to oils which, after saponification, produce a highly colored solution which obscures the recognition of the indicator color change at the end points. Erroneous results are obtained with materials containing appreciable amounts of strong base. Unsaponifiable compounds whose acid or base dissociation constants in water are smaller than 10^{-9} are not detectable and do not interfere; salts react if their hydrolysis constants are greater than 10^{-9}.

Definitions

2. (*a*) *Saponification Number.*—The number of milligrams of potassium hydroxide solution consumed by 1 g of sample under the conditions of the test.

NOTE 1.—The saponification number includes both strong and weak acid constituents that react under the conditions of the test.

(*b*) *Strong-Acid Saponification Number.*—The number of milligrams of potassium hydroxide consumed by the strong-acid constituents in 1 g of sample under the conditions of the test.

(*c*) *Base Saponification Number.*—The number of milligrams of potassium hydroxide equivalent to the excess basic constituents present after saponifying 1 g of sample under the conditions of the test.

Principle of Method

3. This method resolves the acidic and basic constituents of petroleum products into groups having weak-acid,

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[1] This proposed method is under the jurisdiction of the ASTM Committee D-2 on Petroleum Products and Lubricants. Published as information, June, 1951.

strong-acid, and base-ionization properties, provided the dissociation constants of the more strongly acid components are at least 1000 times those of the next weaker group. Total strong and weak-acid constituents are changed to the potassium salts by saponification with KOH. Titration to the first end point with HCl, using para-xylenol blue indicator, neutralizes all KOH added in excess of that used in saponification. The difference between this excess and the total KOH added is a measure of the total strong and weak-acid constituents, and is used to calculate the saponification number. The weak-acid constituents, whose potassium salts have been formed by the saponification, are hydrolyzed to form weak acids and hydroxide ions. Since these weak acids have low ionization constants, an excess of hydroxyl ions is produced, making the solution slightly basic. Titration with standard HCl to the second para-xylenol blue end point, then, is a typical weak base-strong acid titration, with the end point occurring at a pH of about 4. The amount of HCl used to titrate from the first to the second end point is a measure of the weak-acid constituents, and is equivalent to the KOH consumed in their saponification. The difference between the KOH consumed by the total sample and that consumed by the weak-acid constituents is a measure of the strong-acid constituents, and is used to calculate the strong-acid saponification number (Note 2).

NOTE 2.—The difference between the saponification number and the strong-acid saponification number also expresses the amount of weak-acid constituents present or formed during the test.

If titration to the second end point requires an amount of HCl greater than the equivalent amount of KOH added for the original saponification, the oil has basic properties. The number of milligrams of KOH equivalent to the excess basic constituents present in 1 g of sample is the base saponification number.

Outline of Method

4. The sample, dissolved in a mixture of benzene and isopropyl alcohol, is saponified by refluxing for 2 hr with an excess of KOH. The excess KOH is titrated at room temperature with alcoholic HCl, using para-xylenol blue indicator solution to detect both the total saponification end point (blue-green to yellow), and the strong-acid (or base) saponification end point (yellow to red). A "blank" determination is carried along with the test and titrated in the same manner as that used for the sample.

Apparatus

5. (*a*) *Buret*, of 50-ml capacity, graduated in 0.10-ml divisions, calibrated with an accuracy of ±0.05 ml.

(*b*) *Erlenmeyer Flask*, standard form, narrow mouth, of 500-ml capacity, made of chemically resistant glass.

(*c*) *Reflux Condenser*.—A suitable immersion type condenser, made to fit loosely the top of the 500-ml Erlenmeyer saponification flask, and made of chemically resistant glass. It shall conform to the dimensions shown in Fig. 1, except that the manner of attaching the water inlet and outlet ports may be modified if desired.

(*d*) *Optional Saponification Reflux Apparatus:*

(*1*) *Reflux Condenser*.—Allihn-type reflux condenser, made of chemically resistant glass, with a ground-glass water-cooled 24/40 standard taper joint.

(*2*) *Erlenmeyer Flask*.—A 500-ml, chemically resistant glass,[2] narrow-mouth Erlenmeyer flask, with a 24/40 standard taper joint.

(*e*) *Hot-Plate*.—A hot-plate, heated by electricity or steam, that will maintain a 50 per cent mixture of benzene and isopropyl alcohol, contained in a saponification vessel, at active boiling temperature so that an appreciable reflux occurs.

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[2] Pyrex glass has been found satisfactory for this purpose.

Reagents

6. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.

(*a*) *Benzene*, cp, anhydrous.

(*b*) *Hydrochloric Acid Solution, Alcoholic* (*standard 0.2 N*).—Mix 18 to 19 ml of HCl (sp gr 1.18) with 1 liter of isopropyl alcohol (99 per cent). Standardize against Na_2CO_3 frequently enough to detect normality changes of 0.0005 *N*. Accurately weigh 0.325 g of Na_2CO_3 into an Erlenmeyer flask and dissolve in 190 to 200 ml of distilled water. Add 3 to 4 drops of methyl orange indicator, and titrate to a salmon color.

Calculate the normality of the HCl solution as follows:

$$\text{Normality} = \frac{W}{V \times 0.0530}$$

where:

W = grams of Na_2CO_3 used,
V = milliliters of HCl used in the titration, and
0.0530 = milliequivalent weight of Na_2CO_3.

(*c*) *Isopropyl Alcohol* (*99 per cent*), conforming to ASTM Specifications D 770 for Isopropyl Alcohol.[3]

(*d*) *Methyl Orange Indicator Solution.*—Dissolve 0.1 g of methyl orange in 100 ml of distilled water.

(*e*) *Para-Xylenol Blue Indicator Solution.*[4]—Dissolve 0.1 g of *p*-xylenol-sulfonphthalein (para-xylenol blue) in 100 ml of isopropyl alcohol.

(*f*) *Phenolphthalein Indicator Solution.*—Dissolve 1 g of phenolphthalein in 100 ml of isopropyl alcohol.

(*g*) *Potassium Hydroxide Solution, Alcoholic* (*standard 0.2 N*).—Add 12 g of KOH to approximately 1 liter of isopropyl alcohol (99 per cent) contained in a 2-liter Erlenmeyer flask. Boil the mixture gently for 10 to 15 min, stirring to prevent the solid from forming a cake on the bottom. Cool to room temperature, stopper the flask, and allow to stand for several hours; then filter the supernatent liquid through a fine sintered-glass crucible. Avoid unnecessary exposure to CO_2 during the filtration. Store the solution in a chemically resistant dispensing bottle out of contact with cork, rubber, or saponifiable stopcock lubricant, and protected against carbon dioxide absorption by a guard tube containing soda lime or soda asbestos (Ascarite).

Standardize against potassium acid phthalate ($KHC_8H_4O_4$) frequently enough to detect normality changes of 0.0005 *N*. Accurately weigh 1.6 g of dried potassium acid phthalate in a 250-ml Erlenmeyer flask and dissolve in 125 ml of carbon dioxide-free distilled water. Add 3 to 4 drops of phenolphthalein indicator solution and titrate with the alcoholic KOH solution until the solution turns just pink.

Calculate the normality of the KOH solution as follows:

$$\text{Normality} = \frac{W}{V \times 0.2042}$$

where:

W = grams of potassium acid phthalate used,
V = milliliters of KOH used in the titration, and
0.2042 = milliequivalent weight of potassium acid phthalate.

(*h*) *Titration Solvent.*—Add 500 ml of benzene and 5 ml of water to 495 ml of isopropyl alcohol (99 per cent).

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[3] 1949 Book of ASTM Standards, Part 4.

[4] Solid *p*-xylenolsulfonphthalein can be obtained from Hartman-Leddon Co., Inc., 5821 Market St., Philadelphia 39, Pa.

Procedure

7. (*a*) *Sample Size.*—Determine the size of sample according to the expected saponification number, as indicated in the following table:

Type of Sample	Saponification No.	Size of Sample, g	Sensitivity of Weighing, g
Oils	0 to 5	20.0 ± 2.0	0.05
	5 to 30	5.0 ± 0.5	0.02
	30 to 80	2.0 ± 0.2	0.01
	80 to 200	0.8 ± 0.1	0.005
	over 200	0.5 ± 0.05	0.002
Fats and Fatty Oils	below 200	0.8 ± 0.1	0.002
	over 200	0.5 ± 0.05	0.001

NOTE 3.—If the appropriate sample size is too large to form a homogeneous solution, or so dark that the end point is obscured, a smaller sample may be taken. It must be recognized, however, that the precision and accuracy may suffer accordingly.

(*b*) *Saponification.*—Weigh the sample, of size specified in Paragraph (*a*), into a 500-ml Erlenmeyer flask, and add 40 to 45 ml of cp benzene. From a 50-ml buret, add 40.0 ± 0.05 ml of the 0.2 *N* alcoholic KOH solution, allowing 60 to 80 sec to elapse between adding the first 39.5 ml and the final 0.5 ml. Connect the flask to the condenser, heat to boiling on a hot plate, and reflux for 110 to 120 min.

(*c*) *Preparation for Titration.*—Disconnect the condenser and wash the exposed condenser and flask surface with 10 to 20 ml of solvent, catching the washings in the flask. If there is no evidence of a precipitate clinging to the sides of the reaction flask, cover it with a small inverted beaker, cool to room temperature, and titrate as directed in Paragraph (*d*). If there is any evidence of a precipitate clinging to the sides of the flask, quickly, while the contents are still hot, transfer the liquid phase by decantation into a clean 500-ml Erlenmeyer flask. Rinse the original saponification flask by decantation with three 5-ml portions of titration solvent. Add the rinsings to the second flask, cover with a small inverted beaker, and cool the flask and contents to room temperature. As soon as possible after the decantations, add 100 ml of CO_2-free distilled water to the original saponification flask and heat just to boiling. Cover the flask with a small inverted beaker and cool to room temperature. Titrate, within 2 hr, the nonaqueous solution as directed in Paragraph (*d*), and the aqueous solution as directed in Paragraph (*e*).

(*d*) *Titration of Nonaqueous Solution.*—Fill the buret with 0.2 *N* alcoholic HCl solution and place the buret in position so that the tip may extend into the neck of the flask while the flask is being swirled or shaken. Add 6 ± 1 ml of the para-xylenol blue indicator solution to the contents of the flask. From the buret add acid, cautiously but rapidly, as long as no momentary color change takes place on the surface of the solution. Upon approaching the first end point (blue-green to yellow), gradually decrease the size of the increments of acid until the color change takes place with the addition of 0.02-ml increments. Record the volume of acid required to reach this first color change as *A* (Note 4). Continue the titration on the same solution in like manner until the second color change (yellow to red) takes place. Record the volume of acid to reach the second end point as *C* (Note 4).

NOTE 4.—Shake vigorously near both end points, but avoid prolonged shaking which may dissolve appreciable amounts of CO_2 in the solvent. With dark-colored oils, the observation of the end point may be facilitated by viewing the color change through a slight foam produced momentarily by a more violent shaking of the flask, and by observing the titration under a white fluorescent lamp at bench-top level.

An additional aid in determining the end point is the constant addition of titrant at the rate of 1 or 2 drops a second, at the same time continually swirling as the end point is reached.

In this manner the eyes do not "forget" a color or intensity of color as they generally do between periodic additions and shakings. Consider the end points definite if the color change persists for 15 sec or if it is reversed with 1 to 2 drops of standard 0.2 *N* base.

(*e*) *Titration of Aqueous Solution.*—To the aqueous solution, add 3 to 5 drops of phenolphthalein indicator solution and titrate with 0.2 *N* alcoholic HCl solution until the pink color just disappears. Record the volume of acid to reach this phenolphthalein end point as *E*. Add 3 to 5 drops of methyl orange indicator solution and continue titrating until the color of the solution is salmon. Record the volume of acid corresponding to the methyl orange end point as *G*.

NOTE 5.—Shake vigorously near both end points, but avoid prolonged shaking which may dissolve appreciable amounts of CO_2 in the solvent. Consider the end points definite if the color change persists for 15 sec or if it is reversed with one or two drops of standard 0.2 *N* base.

(*f*) *Blank Determinations.*—Make duplicate blank determinations simultaneously with each set of sample determinations, following exactly the procedure used for the sample and using the same quantities of all reagents, but omitting the sample.

Calculations and Report

8. (*a*) Calculate the saponification, strong-acid saponification, and base saponification numbers by means of the following equations:

Saponification number, mg of KOH per g

$$= \frac{(B + F - A - E)\ N \times 56.1}{W}$$

Strong-acid saponification number, mg of KOH

$$\text{per g} = \frac{(D + H - C - G)\ N \times 56.1}{W}$$

Base saponification number, mg of KOH per g

$$= \frac{(C + G - D - H)\ N \times 56.1}{W}$$

where:

A = milliliters of standard HCl solution to neutralize the excess base in titrating the nonaqueous solution to the first para-xylenol blue end point,
B = value corresponding to A for the blank titration,
C = milliliters of standard HCl to neutralize the basic materials in titrating the nonaqueous solution to the second para-xylenol blue end point,
D = value corresponding to C for the blank titration,
E = milliliters of standard HCl solution to neutralize the excess base in titrating the water solution to the phenolphthalein end point,
F = value corresponding to E for the blank titration,
G = milliliters of standard HCl to neutralize the basic materials in titrating the water solution to the methyl orange end point,
H = value corresponding to G for the blank titration,
N = normality of standard HCl solution,
W = g of sample, and
56.1 = equivalent weight of KOH.

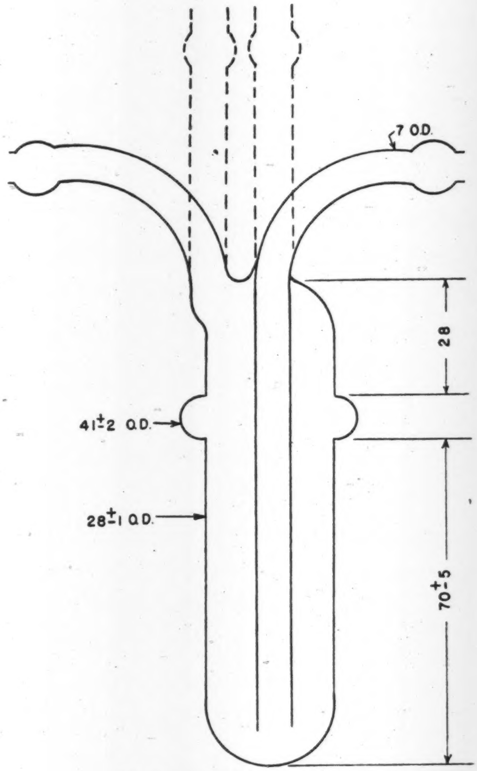
When the saponification does not produce a clinging precipitate, thus making the aqueous titration unnecessary, the terms *E*, *F*, *G*, and *H* are all zero.

(*b*) In the analysis of compounded oils, the fat or fatty oil content can be calculated from the saponification number by means of the following equation:

$$\text{Fat or fatty oil, per cent by wt} = \frac{L - K}{M - K} \times 100$$

where:

K = saponification number of the unblended base stock,
L = saponification number of the compounded oil,
M = saponification number of the unblended fat or fatty oil added, and
100 = percentage factor.



All dimensions in millimeters.

FIG. 1.—Reflux Condenser.

NOTE 6.—The fat or fatty oil content of compounded oils may be calculated only if the sample contains but one saponifiable fat or fatty oil, whose saponification number is known or can be determined from a sample of the unblended fat. If it is known that the base oil does not contain appreciable amounts of nonfatty materials which react with acid or alkali under the conditions of the test, or if the base oil is known to be an uncompounded refined petroleum oil, its saponification number may be considered equal to zero. Otherwise, the saponification number of the base oil must be a known value or a value determined on the unblended stock.

(*c*) Report the calculated values as saponification number, strong-acid saponification number, or base saponification number. Report all negative values for strong-acid or base saponification numbers as zero. Report fat or fatty oil contents as weight per cent fat or fatty oil.

Precision

9. Results should not differ from the mean by more than the following amounts:

Saponification No.	Repeatability	Reproducibility
0.0 to 5.0..........	0.1	0.15
5.0 to 30.0..........	0.4	0.6
30.0 to 80.0..........	1.0	1.5
80.0 to 200..........	3.0	4.5
over 200..............	4.0	6.0

These precision values do not apply to oils that are not completely soluble in the solvent or that color the solvent to such an extent that the end point color change is obscured.

APPENDIX IV

PROPOSED METHOD OF TEST FOR OIL CONTENT OF PETROLEUM WAXES[1]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race Street, Philadelphia 3, Pa.

Scope

1. This method describes a test procedure for the determination of oil in petroleum waxes having a melting point of 105 F or higher, and containing not more than 15 per cent of oil.[2]

NOTE.—This method is essentially the same as ASTM Method D 721 - 51 T, Tentative Method of Test for Oil Content of Paraffin Wax,[3] but it has been modified by a stirring operation in order to extend the procedure to microcrystalline waxes, which may dissolve with difficulty in methylethylketone.[4]

Outline of Method

2. The sample is dissolved in methylethylketone, the solution cooled to −25 F to precipitate the wax, and filtered. The oil content of the filtrate is determined by evaporating the methylethylketone and weighing the residue.

Apparatus

3. The following apparatus[5] is required:

(*a*) *Filter Stick and Assembly*, consisting of a 10-mm diameter medium-porosity sintered glass filter stick, provided with an air pressure inlet tube and delivery nozzle. It is provided with a ground-glass joint to fit a 25 by 170-mm test tube. The dimensions for a suitable filtration assembly are shown in Fig. 1.[6]

NOTE.—A metallic filter stick may be employed if desired. A filter stick made of stainless steel and having a ½-in. disk of medium porosity has been found to be satisfactory.[7] The metallic apparatus is inserted into a 25 by 150-mm test tube and held in place by means of a cork.

(*b*) *Cooling Bath*, consisting of an insulated box with 1-in. holes in the center to accommodate any desired number of test tubes. The bath may be filled with a suitable medium such as kerosine, and may be cooled by circulating a refrigerant through coils, or by using solid carbon dioxide. A suitable cooling bath to accommodate three test tubes is shown in Fig. 2.[6]

(*c*) *Dropper Pipet*, provided with a rubber bulb, and calibrated to deliver 1 ± 0.05 g of molten wax.

(*d*) *Transfer Pipet*, calibrated to deliver 15 ± 0.06 ml.

(*e*) *Air Pressure Regulator*, designed to supply air to the filtration assembly (Section 6(*e*)) at the volume and pressure required to give an even flow of filtrate.

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[1] This proposed method is under the jurisdiction of the ASTM Committee D-2 on Petroleum Products and Lubricants. Published as information, June, 1951.

[2] This method is being used by some laboratories for products of lower melting point and higher oil content.

[3] 1951 Supplement to Book of ASTM Standards, Part 5.

[4] The oil content values obtained on microcrystalline waxes must be considered only arbitrary values where "oil" is defined by the conditions of this test. These values will generally not agree with those obtained by other methods employing different solvents or conditions.

[5] This apparatus may be obtained from Kopp Scientific Laboratory Equipment, Inc., 405 East 62nd St., New York 21, N. Y.

[6] These illustrations of the apparatus are identical with those appearing in Method D 721 - 51 T, see pp. 134 to 138, 1951 Supplement to Book of ASTM Standards, Part 5.

[7] A suitable metal filter stick with designated porosity G may be obtained from the Micro Metallic Corp., 193 Bradford Street, Brooklyn, N. Y.

Either the conventional pressure-reducing valve or a mercury bubbler-type regulator has been found satisfactory. The latter type, illustrated in Fig. 3,[6] consists of a 250-ml glass cylinder and a T-tube held in the cylinder by means of a rubber stopper grooved at the sides to permit the escape of excess air. The volume and pressure of the air supplied to the filtration assembly is regulated to a depth to which the T-tube is immersed in mercury at the bottom of the cylinder. Absorbent cotton placed in the space above the mercury prevents the loss of mercury by spattering. The air pressure regulator is connected to the filter stick and assembly by means of rubber tubing.

(*f*) *Thermometers.*—Oil in Wax Thermometers having the range of −35 to +70 F and conforming to the proposed specifications appearing in the Supplement to Method D 721 - 51 T.[3]

(*g*) *Weighing Bottles*, conical in shape and glass-stoppered, having a capacity of 15 ml.

(*h*) *Evaporation Assembly*, consisting of an evaporating cabinet and connections, essentially as illustrated in Fig. 4,[6] and capable of maintaining a temperature of 95 ± 2 F around the evaporation flasks. Construct the jets with an inside diameter of 4 ± 0.2 mm for delivering a stream of clean, dry air vertically downward into the weighing bottle. Support each jet so that the tip is 15 ± 5 mm above the surface of the liquid at the start of the evaporation. Supply air at the rate of 2 to 3 liters per min per jet, purified by passage through a tube of 1-cm bore packed loosely to a height of 20 cm with absorbent cotton. Periodically check the cleanliness of the air by evaporating 4 ml of methylethylketone by the procedure specified in Section 6(*e*). When the residue does not exceed 0.1 mg, the evaporation equipment is operating satisfactorily.

(*i*) *Analytical Balance*, capable of reproducing weights to 0.1 mg. The sensitivity should be adjusted so that 0.1 mg will deflect the pointer one half division on the pointer scale.

(*j*) *Wire Stirrer.*—A piece of stiff iron or nichrome wire of about No. 20 B & S gage, 250 mm long. A 10-mm diameter loop is formed at each end, and the loop at the bottom end is bent so that the plane of the loop is perpendicular to the wire.

Solvent

4. *Methylethylketone*, conforming to ASTM Specifications D 740, for Methyl Ethyl Ketone,[8] and having a refractive index at 68 F of 1.378 ± 0.002. The residue remaining after the evaporation of 4 ml by the procedure specified in Section 6(*e*) must not exceed 0.1 mg.

Sample

5. If the sample of wax is 2 lb or less, obtain a representative portion by melting the entire sample and stirring thoroughly. For samples over 2 lb, exercise special care to insure obtaining a truly representative portion, bearing in mind that the oil may not be distributed uniformly throughout the sample, and that mechanical operations may express some of the oil.

Procedure

6. (*a*) Melt a representative portion of the sample in a beaker, using a water bath or oven maintained at 160 to 210 F. As soon as the wax is completely melted, thoroughly mix by stirring. Preheat the dropper pipet in order to prevent the solidification of wax in the tip, and withdraw a 1-g portion of the sample as soon as possible after the wax has melted. Hold the pipet in a vertical position, and carefully transfer its contents into a clean, dry test tube previously weighed to the nearest 1 mg (Note 1). Swirl the

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[8] 1949 Book of ASTM Standards, Part 4.

test tube so as to coat the bottom evenly with wax. This permits more rapid solution later. Allow the test tube to cool, and weigh to the nearest 1 mg.

NOTE 1.—The weight of a test tube which is cleaned by means of solvents will not vary to a significant extent. Therefore, a tare weight may be obtained and used repeatedly.

(*b*) Pipet 15 ml of methylethylketone into the test tube and place the latter just up to the level of its contents in a hot water or steam bath. Heat the solvent - wax mixture, stirring up and down with the wire stirrer, until a homogeneous solution is obtained. Exercise care to avoid loss of solvent by prolonged boiling.

NOTE 2.—Very high-melting microcrystalline wax samples may not form clear solutions. Stir until the undissolved material is well dispersed as a fine cloud.

Plunge the test tube into an 800-ml beaker of ice water and continue to stir until the contents are cold. Remove the stirrer. Remove the test tube from the ice bath, wipe dry on the outside with a cloth, and weigh to the nearest 0.1 g.

NOTE 3.—During this operation the loss of solvent through vaporization should be less than 1 per cent. The weight of the solvent is, therefore, practically a constant, and, after a few samples are weighed, this weight, approximately 11.9 g, can be used as a constant factor.

(*c*) Place the test tube containing the wax - solvent slurry in the cooling bath, which is maintained at −30 ± 2 F. During this chilling operation it is important that stirring by means of the thermometer be almost continuous, in order to maintain a slurry of uniform consistency as the wax precipitates. Do not allow the wax to set up on the walls of the cooling vessel nor permit any lumps of wax crystals to form. Continue stirring until the temperature reaches −25 ± 0.5 F.

(*d*) Immerse in the mixture the clean, dry filter stick, which has previously been cooled by placing it in a test tube and holding at −30 ± 2 F in the cooling bath for a minimum of 10 min. Seat the ground-glass joint of the filter so as to make an air-tight seal. Place an unstoppered weighing bottle, previously weighed together with the glass stopper to the nearest 0.1 mg, under the delivery nozzle of the filtration assembly.

NOTE 4.—Take every precaution to insure the accuracy of the weight of the stoppered weighing bottle. Prior to determining this weight, rinse the clean, dry, weighing bottle and stopper with methylethylketone, wipe dry on the outside with a cloth, and place in the evaporation assembly to dry for about 5 min. Then remove the weighing bottle and stopper, place near the balance, and allow to stand for 10 min prior to weighing. Stopper the bottle during this cooling period. Once the weighing bottle and stopper have been dried in the evaporation assembly, lift only with forceps. Take care to remove and replace the glass stopper with a light touch.

(*e*) Apply air pressure to the filtration assembly, and immediately collect about 4 ml of filtrate in the weighing bottle. Release the air pressure to permit the liquid to drain back slowly from the delivery nozzle. Remove the weighing bottle immediately, and stopper and weigh to the nearest 10 mg without waiting for it to come to room temperature. Unstopper the weighing bottle and place it under one of the jets in the evaporation assembly maintained at 95 ± 2 F, with the air jet centered inside the neck, and the tip 15 ± 5 mm above the surface of the liquid. After the solvent has evaporated, which usually takes less than 30 min, remove the bottle, stopper, and place near the balance. Allow to stand for 10 min and weigh to the nearest 0.1 mg. Repeat the evaporation procedure, using a 5-min evaporation period instead of 30 min, until the loss between successive weighings is not over 0.2 mg.

Calculation

7. Calculate the amount of oil in the wax as follows, and if the result is negative report as zero.

Oil in Wax, per cent by weight

$$= \frac{100\,AC}{BD} - 0.15$$

where:

A = weight of oil residue, in grams,
B = weight of wax sample, in grams,
C = weight of solvent, in grams, obtained by subtracting weight of test tube plus wax sample (Section 6(*a*)) from weight of test tube and contents (Section 6(*b*)), and
D = weight of solvent evaporated, in grams, obtained by subtracting weight of weighing bottle plus oil residue from weight of weighing bottle plus filtrate (Section 6(*e*)).

0.15 = average factor correcting for the solubility of wax in the solvent at −25 F.

Precision

8. Results should not differ from the mean by more than the following amounts:

Oil in Wax, per cent	**Repeatability** One Operator and Apparatus	**Reproducibility** Different Operators and Apparatus
Unde 1.........	0.1	0.2
1 and over.......	0.2	0.4

APPENDIX V

PROPOSED METHOD OF TEST FOR KINEMATIC VISCOSITY[1]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

Scope

1. This method describes test procedures for the accurate measurement of kinematic viscosities of newtonian (simple) liquids. Determinations can be made at any temperature at which the liquid is newtonian. Kinematic viscosity values are particularly recommended for calculation of viscosity indices of lubricating oils by ASTM Method D 567.[2]

NOTE 1.—This method has been found useful in measuring the apparent viscosity of nonnewtonian liquids.[3]

NOTE 2.—Many liquids have a temperature range at which they are newtonian. Due to nonhomogeneity at low temperatures, and to instability or decomposition at high temperatures, some liquids become nonnewtonian at low and high temperatures. The point of deviation on a Viscosity–Temperature curve plotted on an ASTM Viscosity–Temperature chart is an indication of the point where the liquid becomes nonnewtonian. A second criterion for detecting nonnewtonian behavior is measuring different rates of shear, such as measuring the viscosity of the liquid in several viscometers all of different capillary diameters.

NOTE 3.—The conversion of Kinematic viscosity to Saybolt universal viscosity is covered by ASTM Method D 446,[2] and to Saybolt furol viscosity by ASTM Method D 666.[2]

Definitions

2. (*a*) *Dynamic or Absolute Viscosity.*—The cgs unit of dynamic viscosity (V_a) is the poise, which has the dimensions grams per centimeter per second.

(*b*) *Kinematic Viscosity* is the quotient of the dynamic viscosity divided by the density, V_a/d, both at the same temperature. The unit of kinematic viscosity (V_k) is the stoke, which has the dimensions square centimeters per second. It is more common in the petroleum industry to use the hundredth part of stoke, centistoke (cs), in expressing the viscosity of a product.

(*c*) *Newtonian* (*simple*) *Liquid* is one in which the rate of shear is proportional to the shearing stress (see ASTM Standard E 24, Definitions of Terms Relating to Rheological Properties of Matter).[2]

Outline of Method

3. The time is measured for a fixed volume of sample, contained in a glass viscometer, to flow through a calibrated capillary under an accurately reproducible head of liquid and at a closely controlled temperature. From the time measured, the kinematic viscosity is calculated. The viscometers are calibrated by using standard oils having viscosities established by reference to water in master (long capillary) viscometers, or by direct comparison with carefully calibrated viscometers.

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[1] This proposed method is under the jurisdiction of the ASTM Committee D-2 on Petroleum Products and Lubricants. Published as information, June, 1951.

This proposed method is a proposed revision of, and is intended to replace, when adopted, the Tentative Method of Test for Kinematic Viscosity (D 445 - 46 T), see p. 186. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

[2] 1949 Book of ASTM Standards, Part 5.

[3] Research Division VII, Section B on Non-Newtonian Liquids, is under the jurisdiction of the ASTM Committee D-2 on Petroleum Products and Lubricants.

Apparatus

4. (*a*) *Master Viscometers,* glass, in which the quantity B/t is a negligible per cent of the quantity Ct as determined in Section 6.[4] This is required because B is not a constant. It varies with Reynolds Numbers.[5] These viscometers are required for basic calibration in which water is the primary standard.

(*b*) *Viscometers,* capillary type, glass, capable of measuring viscosity with an error not greater than specified in Section 9. Apparatus of various designs[6] have been successfully employed and are commercially available and acceptable. The design of the viscometer under specific conditions must be such that the quantity B/t will not exceed 1 per cent of the quantity Ct as referred to in Section 6. For opaque oils it is necessary to use an instrument in which the forward movement of the sample into the clean portion of the tube is observed.

NOTE 4.—To provide a nearly equal overlap between viscometers with adjacent constants it is suggested that a viscometer series having C constants of 1 and 3 times powers of 10 (from 0.003 to 100) be used. Viscometers constructed with C constants of simple integer multipliers (1 or 3) are most adaptable for routine work; that is, C constants of exactly 0.003, 0.01, 0.03, 0.1, 0.3......30.0, 100.0.

(*c*) *Thermometers.*—ASTM Kinematic Viscosity Test Thermometers graduated in Fahrenheit degrees, having ranges as indicated in Table I for commonly specified test temperatures, and conforming to the requirements for these thermometers as prescribed in ASTM Standard E 1[7] and in the Supplement to this method. The use of other thermometric devices is acceptable providing that their accuracy, precision and sensitivity are equal to, or better than, those of the specified thermometers.

NOTE 5.—The ASTM Kinematic Viscosity Thermometers are standardized for conditions of "total immersion," which means immersion to the top of the mercury column, with the remainder of the stem and the expansion chamber at the top of the thermometer exposed to room temperature. If the thermometer is completely submerged in the bath during use, the pressure of the gas in the expansion chamber will be higher than during the standardization, and may cause low readings on the thermometer. In the case of the thermometer for tests at 210 F, the reading may be as much as 0.2 F low. The practice of submerging the thermometer, therefore, is not recommended. If thermometers are completely submerged, corrections for each individual thermometer based on calibration under conditions of complete submergence must be determined and applied.

TABLE I.—ASTM KINEMATIC THERMOMETERS.

Temperature Range, deg Fahr.	Subdivision, deg Fahr	ASTM Thermometer No.
−61 to −29......	0.2	43F - 49T
−31 to +1.......	0.2	[a]
−1 to +33......	0.2	[a]
31 to +65......	0.2	[a]
66.5 to 71.5....	0.1	44F - 49T
74.5 to 79.5. ..	0.1	45F - 49T
97.5 to 102.5....	0.1	28F - 49T
119.5 to 124.5....	0.1	46F - 49T
127.5 to 132.5....	0.1	29F - 49T
137.5 to 142.5....	0.1	47F - 49T
177.5 to 182.5....	0.1	48F - 49T
207.5 to 212.5....	0.1	30F - 49T

[a] See the proposed specifications in the Supplement to this method.

Apply calibration corrections to all thermometer readings. Directions for determining these corrections are given in explanatory notes in ASTM Standard E 1.[7]

(*d*) *Bath.*—A bath suitable for immersion of the viscometer so that the oil resevoir or the top of the capillary, whichever is uppermost, is at least 5 cm below the upper bath level, and with provision for visibility of the instrument and the thermometer. Firm supports for the viscometer shall be provided; or the viscometer may be sealed in as an integral part of the bath. Either a liquid bath with thermostatic regulation and a stirrer or a vapor bath with pressure regulation is permissible. The efficiency of the stirring and the balance between heat losses and heat input must be such that the temperature of the bath medium does not vary by more than ±0.02 F over the length of the viscometer, or from viscometer to viscometer in the various bath positions for temperatures above 60 F. The variation for test temperatures below 60 F must not exceed ±0.05 F. If a vapor bath is used, there must be no temperature gradient over the length of the viscometer greater than that permitted in a liquid bath.

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[4] See M. R. Cannon, "Viscosity Measurement, Master Viscometers," *Industrial and Engineering Chemistry,* Analytical Editions Vol. 16, November 15, 1944, p. 708.

[5] See James Donald Bell, "The Variation of the Kinetic Energy Correction with Reynolds Number," Penn State College (1947).

[6] See list of references at end of this method to papers describing viscometers of various designs. Section A of Research Division VII is preparing a new appendix which will contain specific operational directions and instrument specifications for each viscometer for which there is a published reference. ASTM Method D 445, Tentative Method of Test for Kinematic Viscosity, Appendix, p. 901 of 1949 Book of ASTM Standards, Part 5, describes three instruments.

[7] 1951 Supplement to Book of ASTM Standards, Part 5.

(*e*) *Timer.*—Stop watch or other spring-activated timing device, graduated in divisions of 0.2 sec or less, and accurate to within 0.05 per cent when tested over not less than a 10-min period. Such errors may occur in timing devices actuated by electrical synchronous motors, driven by most public power systems, which are intermittently and not continuously controlled. Such electrical timing devices shall be used only on electrical circuits of continuously controlled frequency. Frequency-controlled devices of suitable capacity for laboratory purposes, accurate to within 1 part in 10,000, should be used.

NOTE 6.—Time signals as broadcast by the National Bureau of Standards,[8] Station WWV, at 2.5, 5, 10, 15, 20, 25, 30, and 35 megacycles are a convenient and primary standard reference for calibrating timing devices. The signals are broadcast 24 hr daily. Experimental Station WWVH, broadcasting the same signals from Hawaii, may be received better by those west of the Rocky Mountains on 5, 10, and 15 megacycles. Due to propagation conditions, all frequencies might not be available locally. It is, therefore, desirable to try each frequency until one of satisfactory volume is received.

Reference Materials

5. (*a*) *Freshly Distilled Water* is the primary standard for the calibration of Master Viscometers and secondary standard oils. The values given in Table II are not precise to the last significant figure, but these values are designated to establish a standard until better values are known.

TABLE II.—KINEMATIC VISCOSITY OF WATER.[9]

At 68.00 F (20.00 C)...	1.007 centistokes
At 100.00 F (37.78 C)...	0.689 centistokes

(*b*) *API Liquid Viscosity Standards*, having the approximate kinematic viscosities shown in Table III. Certified kinematic viscosity values established by annual cooperative tests are supplied with each sample, which are identified with a year date following the Greek letter designation.

TABLE III.—API VISCOSITY STANDARDS.

API Viscosity Oil Standards	Approximate Kinematic Viscosity, centistokes		
	At −40 F	At 100 F	At 210 F
Alpha............	...	66	8
Beta.............	...	365	27
Gamma...........	85	3	...

NOTE 7.—The viscosity standards are available in 8-oz containers, price $2.50 each, and in 1-qt containers, price $5.00 each. Purchase orders should be addressed to Lacey Walker, Secretary, American Petroleum Institute, 50 West 50th Street, New York 20, New York. Remittance, payable to the American Petro-

leum Institute, with the order, will expedite shipment. Shipment will be made by express collect to points within the United States. The American Petroleum Institute will not accept orders requiring shipment outside the United States. Purchasers outside the United States may order oils shipped to their designated agents in this country. The API will accept no further responsibility than delivery to such agent.

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[8] See W. D. George, "Production of Accurate One-Second Time Intervals," *Journal of Research*, Nat. Bureau Standards, Vol. 21, September, 1938, pp. 367–373. (*RP* 1136.)

[9] The values are taken from the work of E. C. Bingham and R. F. Jackson, *Bulletin*, Nat. Bureau Standards, Vol. 14, pp. 59–86 (1916).

(*c*) *NBS Liquid Viscosity Standards*, having the approximate kinematic viscosities shown in Table IV. Accurate viscosity values are reported with each sample at each temperature indicated.

TABLE IV.—NBS VISCOSITY STANDARDS.

NBS Viscosity Oil Standards	Approximate Kinematic Viscosity, centistokes			
	At 68 F	At 77 F	At 100 F	At 104 F
D	2.6	2.3	1.9	...
H	9.1	7.8	5.5	...
I	14	12	8.1	...
J	25	21	13	...
K	48	38	22	...
L	110	84	43	...
M	320	230	110	...
N	1600	1100	460	...
OB	39000	25000	...	6900

NOTE 8.—The NBS Viscosity Standards are available only as 1-pt samples. The price per sample FOB Washington, D. C., is $10.00 for standards D through N, and $25.00 for standard OB. An additional charge of $10.00 per sample per temperature is made for viscosity determinations at other temperatures in the range 68 to 212 F on oils D through N. For standard OB, the extra charge for special temperature points is $16.00 per temperature. Purchase orders should be accompanied by remittance, payable to the Treasurer of The United States of America, and should be addressed to the National Bureau of Standards, Washington 25, D. C.

(*d*) *Other Reference Liquid Viscosity Standards*, for the calibration of routine viscometers, may be established by choosing suitable stable oils covering the desired viscosity range, or by blending suitable stable oils and determining the viscosities of these oils in the viscometer series calibrated as described in Section 6(*a*). Store the reference standards in light-resistant containers with oil-resistant closures and away from light and heat.

Calibration

6. Calibrate viscometers in accordance with one of the methods described in the following paragraphs:

(*a*) *Basic Calibration.*—Determine the efflux time, to the nearest 0.1 sec, of freshly distilled water in a master viscometer in which water will have a minimum efflux time of 200 sec at test temperature. Using the values given in Table II, calculate the viscometer constant, C, as follows:

$$C = \frac{V_k}{t} \quad \text{.............} (1)$$

where:

V_k = kinematic viscosity of water in centistokes at test temperature, and

t = efflux time in seconds.

NOTE 9.—Differences in the surface tensions of water and oil require that a correction for this variable be subtracted from the efflux time of the water. Calculate the surface tension correction as follows:

$$\Delta H = \left(\frac{2}{g}\right)\left(\frac{S_w}{d_w} - \frac{S_o}{d_o}\right)\left(\frac{1}{r_1} - \frac{1}{r_2}\right) \ldots (2)$$

Surface tension correction, per cent

$$= \frac{\Delta H \times 100}{H} \, .. (3)$$

where:

g = gravitational constant (980),

S_w = surface tension of water (72), in dynes per cm,

S_o = surface tension of oil, in dynes per cm,

d_w = density of water, in g per ml,

d_o = density of oil, in g per ml,

r_1 = average working radius of efflux bulb in cm,

r_2 = average working radius of lower reservoir in cm,

H = average driving head, and
ΔH = change in average driving liquid head in viscometer due to difference in surface tension of water and oil.

The measured efflux time for oil is reduced by this percentage for those viscometers in which the upper meniscus is smaller in diameter than the lower meniscus and the measured efflux time should be increased by this percentage in those viscometers where the upper meniscus is larger than the lower meniscus.

Determine the viscosity of a hydrocarbon more viscous than water in the same viscometer and utilize this in turn to calibrate, as described above, a second Master Viscometer of larger capillary diameter, in which the efflux time of the hydrocarbon exceeds 200 sec. Calibrate other master viscometers of successively larger capillary diameter in a similar manner, using in each case capillary diameters in which the flow time of the calibrating oils exceeds 200 sec. It is advisable to use a minimum of two oils in each viscometer by this procedure. In order to avoid accumulative error in this "step-up" procedure, check each size viscometer against the viscometer second below in the series by means of additional oils.

(*b*) *Calibration of Routine Viscometers by Means of Oil Standards.*—Measure in the viscometer the efflux time of an API or NBS Viscosity Standard or other reference liquid (Section 5 (*d*)). The minimum efflux time for any reference oil in any tube being calibrated should not be less than 200 sec. Determine the viscometer coefficient B, applicable to viscometers having small capillaries and C constants of 0.05 or less, by measuring the efflux time of two oils differing at least five-fold in viscosity and flow time. The lowest value must exceed 200 sec. Calculate B as follows:

$$\text{Viscometer coefficient } B = \frac{t_1 t_2}{t_2^2 - t_1^2}\,(V_2 t_1 - V_1 t_2)\,..\,(4)$$

where:

t_1 = efflux time (200 sec per min) for an oil having a kinematic viscosity of V_1, and
t_2 = efflux time for an oil having a kinematic viscosity of V_2 (about five times greater than V_1).

NOTE 10.—The kinetic energy coefficient B is not a constant in capillary viscometers. It increases with increasing Reynolds Numbers.[10] The assumption that B is a constant will lead to increasing errors as the viscosity of the test liquid decreases. The correction becomes negligible when viscosities exceed 10.0 centistokes. For some viscometers this correction is zero.

Calculate the viscometer constant, C, as follows:

$$\text{Viscometer constant } C = \frac{V + \frac{B}{t}}{t}\,\ldots(5)$$

where:

V = kinematic viscosity in centistokes, of the calibrating oil,
t = efflux time in sec, and
B = viscometer coefficient (Eq. 4), the kinetic energy correction characteristic of the viscometer used.

(*c*) *Calibration of Routine Viscometers by Means of Instrument Standards.*—By simultaneous comparison in the same bath, using the same oil, compare viscometers directly with other viscometers calibrated as described in Paragraph (*a*) or Paragraph (*b*). Calculate the viscometer constant C_1 as follows:

$$\text{Viscometer constant } C_1 = \frac{t_2 \times C_2}{t_1}\,..\,(6)$$

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[10] See G. Reynolds, "An Investigation of the Circumstances Which Determine Whether the Motion of Water Shall be Direct or Sinuous, and of the Law of Resistance in Parallel Channels," *Philosophical Transactions*, London, Chapt. 174, p. 935; E. Bingham, "Fluidity and Plasticity," *International Chemical Series*, Chapt. 3, p. 40; and Brown and Associates, "Unit Operations," Chapt. 6, p. 68, Chapt. 7, p. 80, and Chapt. 12, p. 138, J. Wiley and Sons (1950).

where:

C_1 = viscometer constant being calculated,
t_1 = efflux time in sec, of calibrating oil in viscometer being calibrated,
C_2 = viscometer constant C of standard instrument, and
t_2 = efflux time in sec, of calibrating oil in standard viscometer.

NOTE 11.—Direct comparison calibration is best accomplished by using viscometers of approximately the same capillary diameter.

NOTE 12.—Correction to constant C for viscometers in which the liquid volume is measured at other than test temperature. The constant, C, for the modified Ostwald type viscometers varies according to, and can be calculated as follows:

$$CT_2 = CT_1 \frac{\times 1 - (V_2 - V_1)}{0.785\ \mathrm{H}\ \mathrm{d}^2} \quad ..(7)$$

where:

CT_2 = viscometer constant at T_2, the temperature for which the constant is being calculated,
CT_1 = viscometer constant at T_1, the temperature at which the constant is known,
V_1 = total volume of liquid in the viscometer in ml at T_1,
V_2 = total volume of liquid in the viscometer in ml at T_2,
H = fluid head in centimeters, and
d = inside diameter of the lower reservoir in centimeters.

The approximate relationship between constants for different temperatures is as follows:

Calibration constant at 210 F (98.89 C) = 0.996 × calibration constant at 100 F (37.78 C)
Calibration constant at 130 F (54.44 C) = 0.999 × calibration constant at 100 F (37.78 C)
Calibration constant at 60 F (15.56 C) = 1.001 × calibration constant at 100 F (37.78 C)

Procedure

7. The specific details of operation and their order of continuity vary considerably for the various types of viscometers listed in the Bibliography, and references to those details in the literature will be necessary. In all cases, however, follow the general procedure given below:

(*a*) Filter a portion of the oil sample through a 200-mesh screen or other suitable filter to remove solid particles, water or lint. For work at low temperatures remove water with a suitable desiccant.

(*b*) Select a clean, dry viscometer which will give an efflux time greater than 200 sec.

NOTE 13.—Between successive determinations, clean the viscometer thoroughly by rinsing with an appropriate, completely volatile solvent. Whenever necessary, clean the instrument with chromic acid, rinse thoroughly with distilled water, cp acetone, and dry with clean, dry air.

(*c*) Charge the viscometer in the manner dictated by the design of the instrument, this operation being in conformity with that employed when the instrument was calibrated.

(*d*) Provide the viscometer with a firm mounting which will eliminate disturbances in its position when it is in the bath. With the viscometer immersed in the bath so as to agree with procedure outlined in Section 4 (*d*), make certain the viscometer is vertical when compared with a plumb line observed from two directions about 90 deg apart. Maintain the bath at test temperature within ±0.02 F for test temperatures of 60 F or above, and within ±0.05 F for test temperatures below 60 F. Apply the necessary corrections, if any, to all thermometer readings.

(*e*) Allow the charged viscometer to remain in the bath long enough to reach test temperature. Since this time will vary for the different instruments and for different temperatures, establish a safe temperature equilibrium time by trial. Where the design of the viscometer requires it, adjust the volume of the test sample after the sample has reached temperature equilibrium.

(*f*) When the temperature of the test is below the dew point, fit loosely packed drying tubes onto the open end of the capillary to prevent water condensation in the capillary. There must be no resistance to the flow of air created by the drying tubes.

NOTE 14.—A convenient arrangement for drying tubes is attained by connecting them to both the end of the venting tube and the end of the capillary arm of the viscometer, and connecting them together, at a point between the desiccant and the viscometers, with a large-bore stopcock. The assembly of the drying tubes and the stopcock will form an "H", with the stopcock being in the crossbar of the "H". Air movement during the test will be through the open stopcock and not through the drying tubes.

(*g*) Use vacuum or pressure to adjust the head of the test portion to a position in the capillary arm of the instrument about 5 mm beyond the first timing mark.

(*h*) Measure, in seconds to within 0.1 sec, the time required for the meniscus to pass from the first timing mark to the second. If this efflux time is less than 200 sec, select a smaller capillary viscometer and repeat the operation. Repeat the test and use the average of these two determinations, if they agree within the limits of Repeatability (Section 9), for calculating the kinematic viscosity. If separate viscometers are used, the values obtained must agree within the same limits.

Reporting Results

8. Calculate the kinematic viscosity in centistokes by the following equation:

Kinematic Viscosity, in centistokes,

$$= Ct - \frac{B}{t} \quad \text{. . (8)}$$

where:

C = principal viscometer constant determined as described in Section 6,
t = observed efflux time in seconds, and
B = instrument coefficient as determined and described in Section 6.

For viscometers having large-diameter capillaries and C constants greater than 0.05, use the following equation:

Kinematic viscosity, in centistokes $= Ct$. . . . (9)

where:

C and t are as defined above.

Precision

9. Results should not differ from the mean by more than the following percentages of the mean.

	Repeatability One Operator and Apparatus	**Reproducibility** Different Operators and Apparatus
Test temperature below 60 F.	0.5	1.0
Test temperature 60 F or above.	0.1	0.2

NOTE 15.—Precision within the above limits is not attainable on opaque oils, unless the reverse-flow type instrument is used.

REFERENCES

Widely used and acceptable viscometers are described in the following articles:

L. Ubbelohde, "The Simplest and Most Accurate Viscometer and Other Instruments with Suspended Level," *Journal Institute Petroleum Technologists* (London), Vol. 19, p. 376 (1933); also Oel and Kohle, Vol. 12, p. 949 (1936).

L. Ubbelohde, "The Suspended Level Viscometer," *Journal Institute Petroleum Technologists* (London), Vol. 22, p. 37 (1936).

L. Ubbelohde, "The Principle of the Suspended Level Applications to the Measurement of Viscosity," *Industrial and Engineering Chemistry*, Analytical Edition, Vol. 9, p. 85 (1937).

L. Ubbelohde, "The Viscometer with Suspended Level (The Eliminations of the Influence of Surface Tension by the Suspended Level Spheric Level)," *Journal Institute Petroleum Technologists* (London), Vol. 23, No. 165, p. 427 (1937).

M. R. Cannon and M. R. Fenske, "Viscosity Standardization of Petroleum Lubricating Fractions," *Oil and Gas Journal*, Vol. 33, p. 52 (1935).

M. R. Cannon and M. R. Fenske, "Simple Viscosimeters and Auxiliary Equipment for the Accurate Measurement of Viscosity in Fundamental Units," *Oil and Gas Journal*, Vol. 34, p. 45 (1936).

M. R. Cannon and M. R. Fenske, "Viscosity Measurement," *Industrial and Engineering Chemistry*, Analytical Edition, Vol. 13, p. 299 (1941).

M. R. Cannon, "Viscosity Measurement, Master Viscometers," *Industrial and Engineering Chemistry*, Analytical Edition, Vol. 16, Nov. 15, 1944, p. 708.

E. L. Ruh, R. W. Walker and E. W. Dean, "The S.I.L. Viscosimeter," *Industrial and Engineering Chemistry*, Vol. 13, p. 346 (1941).

E. H. Zeitfuchs, "An Accurate Viscosimeter for Refinery Control Laboratories," *Proceedings American Petroleum Institute*, Vol. 20 (III), (1939).

Ogden FitzSimons, "A Rapid Precision Viscosimeter," *Industrial Engineering and Chemistry*, Analytical Edition, Vol. 7, p. 345 (1935).

M. R. Cannon and M. R. Fenske, "Viscosity Measurement," *Industrial and Engineering Chemistry*, Analytical Edition, Vol. 10, p. 297 (1938).

E. H. Zeitfuchs, "Kinematic Viscometer for Opaque and Very Viscous Liquids," *Oil and Gas Journal*, Jan. 12, 1946, p. 99.

(See Supplement on p. 328)

SUPPLEMENT

For use with this method the following proposed specifications for three Kinematic Viscosity Thermometers, which were prepared by Subcommittee 17 on Thermometers of ASTM Committee E-1 on Methods of Testing at the request of Committee D-2, are published as information only:

Name	ASTM Kinematic Viscosity	ASTM Kinematic Viscosity	ASTM Kinematic Viscosity
Immersion	total	total	total
Temperature Range	−31 to +1 F	−1 to +33 F	+31 to 65 F
Ice Point:			
Range graduated	+31 to +33 F	......	......
Subdivisions	0.2 F	......	......
Bottom of bulb to +32 F	320 to 375 mm	......	......
Subdivisions	0.2 F	0.2 F	0.2 F
Longer Graduation Lines at Each	1 F	1 F	1 F
Graduations Numbered at Each Multiple of	2 F starting at −30 F	2 F starting at 0 F	2 F starting at 32 F
Scale Error at	any point	any point	any point
when Standardized, shall not exceed	0.2 F	0.2 F	0.2 F
Standardization at	Ice point[a] and at −30, −20, −10, 0 F[b]	Ice point[a] and at 0, 10, and 20 F[b]	Ice point[a] and at 40, 50, and 60 F[b]
Total Length	400 to 412 mm	325 to 335 mm	325 to 335 mm
Stem Diameter	7.0 to 8.0 mm	6.5 to 7.5 mm	6.5 to 7.5 mm
Bulb Diameter	6.0 to 7.0 mm	not less than 6.0 mm not greater than stem	
Bulb Length	30 to 40 mm	30 to 40 mm	30 to 40 mm
Contraction Chamber:			
Bottom of chamber to bottom of bulb, min.	290 mm[c]	..	..
Top of chamber to bottom of bulb, max.	310 mm[c]	..	..
Bottom of Bulb to Graduation			
Line at	−31 F	−1 F	+31 F
Distance	60 to 90 mm	60 to 90 mm	60 to 90 mm
Top of Thermometer to Graduation Line at	+1 F	33 F	65 F
Distance	126 to 156 mm	30 to 60 mm	30 to 60 mm
Expansion Chamber shall permit heating to	220 F[c]	220 F[c]	220 F[c]
Top Finish	plain	glass ring	glass ring
Special Inscription on Thermometer	ASTM Kin Vis	ASTM Kin Vis	ASTM Kin Vis
Marking on Case	ASTM Kinematic Viscosity Thermometer −31 to +1 F	ASTM Kinematic Viscosity Thermometer −1 to +33 F	ASTM Kinematic Viscosity Thermometer +31 to 65 F

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[a] See Explanatory Note on Ice Point Determination of Glass Thermometers which appears in the Tentative Method of Testing and Standardization of Etched-Stem Liquid-in-Glass Thermometers (E 77 – 49 T), see 1949 Book of Standards, Part 5.

[b] For conditions of total immersion corrections expressed to the nearest 0.02 F, and such corrections shall be added to the observed reading.

[c] The length of unchanged capillary between nearest graduation mark and expansion or contraction chamber shall be not less than 10 mm.

APPENDIX VI

PROPOSED METHOD OF TEST FOR MOTOR AND RESEARCH OCTANE NUMBERS OF SMALL SAMPLES[1]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

INTRODUCTION

In June, 1949, a study group, known as the "Micro Method Study Group," was formed under Section C of DCC-ASTM to recommend a procedure for the testing of small samples by the Research and Motor methods. Generally, sample volume of at least 450 ml is considered necessary for each of these methods, but this amount, particularly in research work, often means costly repetition of experiments to build up a supply sufficient for the engine tests. A number of techniques were being used in individual laboratories to test smaller samples,[2] and the study group was asked to review them and, if possible, combine their best features or develop new methods and apparatus. After trying different fuel systems and instrumentation, tests of a number of unknown samples in several laboratories indicated that it is possible to determine octane numbers on samples of 50-ml volume with reasonable accuracy by the following method, using either of two fuel systems. The principle involved in the method is that of reducing sample "hold up" in the fuel system and obtaining a fast knockmeter response during the time the fuel is passing through its air-fuel ratio for maximum knock. Tests indicate that the two fuel systems give about equal precision.

Scope

1. In determining the octane numbers of small samples, the ASTM-CFR Engine (Research or Motor) is used. The procedures for checking the engine are the same as those for ASTM Method D 357, Standard Method of Test for Knock Characteristics of Motor Fuels by the Motor Method,[3] and ASTM Method D 908, Standard Method of Test for Knock Characteristics of Motor Fuels by the Research Method,[3] and only differences in apparatus or departures from these methods are given.

Outline of Method

2. To reduce the quantity of fuel required, a small float bowl, or a small pipet, is attached to one of the fuel tanks of the carburetor, and the fuel fed directly to a special horizontal jet, by-passing the sight glass. If a small float bowl is used, the regular fuel tank is made more easily adjustable by means of a crank on the knob of the fuel-tank adjusting screw. Thus, the fuel level may be changed in a slow, continuous motion, causing the air-fuel ratio to pass through its value for maximum knock. If a pipet is used, it can be raised or lowered also by means of the fuel-tank adjusting screw. With a given setting of this adjusting screw, the fuel level changes as the fuel is consumed by the engine, and passes through its

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[1] This proposed method is under the jurisdiction of the ASTM Committee D-2 on Petroleum Products and Lubricants. Published as information, June, 1951.

[2] See "Report on Rating Small Samples of Motor Gasolines," *C.R.C. Report No. F-22A-948;* and "Micro-Determination of Octane Numbers," *Houdry Process Laboratory Report S.G.-49.*

[3] 1949 Book of ASTM Standards, Part 5.

air–fuel ratio for maximum knock. The knock is indicated by a regular knockmeter in connection with the Detonation Meter Model 501 equipped with a six-position, long- to short-time-constant switch.[4] Thus the response or sensitivity of the meter may be varied to suit the engine. The response should be such that it is in step with the change in knock intensity due to change in air–fuel ratio. Maximum readings of the knockmeter are observed on the sample and reference fuels. The reference fuels should bracket the sample in the usual manner and the octane number is determined by interpolation.

Sample Size

3. Samples of 50-ml volume are required for tests by either the Research or Motor method. In cases where the octane number of the sample can be predicted within ±10 octane numbers, the amount of sample may be reduced to 40 ml. A preliminary estimate as to the octane number is always helpful, as it allows presetting the compression ratio within closer limits, thus saving fuel or permitting more fuel for the test proper.

SMALL FLOAT BOWL FUEL SYSTEM

Apparatus

4. The fuel system,[5] modified in accordance with Fig. 1, shall conform to the following requirements:

(*a*) *Jets.*—A special horizontal jet, drill size 0.027 in. for the Motor method, and drill size 0.031 in. for the Research method. These sizes are approximate, and minor adjustments may be necessary (as in ASTM methods) for individual engines, to bring the majority of maximum-knock float levels within 0.8 to 1.8 in. from the top of the vertical jet. The horizontal jet plug and the horizontal jet which serve the right-hand bowl of the carburetor, are removed. These are replaced by the special jet. Details of the jet are shown in Fig. 3.

(*b*) *Fuel Reservoir.*—The fuel reservoir is made of glass, and has a capacity of approximately 50 ml. A portion of a filtering funnel found in most laboratories is suitable. It is supported by a clamp on a bracket which is attached to the right-hand fuel tank of the carburetor.

(*c*) *Bracket.*—The bracket is drilled with three holes, as shown in Fig. 3, such that the two outside holes correspond with the holes in the carburetor fuel-tank body. Standard fuel-tank-gage gaskets are placed on each side of the bracket before attaching the fuel-tank sight-glass gage. In this manner the modified fuel system may be left on the carburetor permanently, without interference with normal operation.

(*d*) *Float Bowl.*—A small float bowl, holding approximately 3 ml, is mounted on the bracket. A Zenith carburetor, Model No. 59-B-3, 9470 Lawson No. 22267, makes a ready substitute, but any other float bowl of the correct capacity may be used. Actually only the float bowl is used in the fuel system, but the whole carburetor is mounted on the bracket by means of two lock nuts on the air tube. The needle valve of the carburetor is kept closed and locked. A $\frac{1}{8}$-in. tube connector is inserted in the inlet to the float chamber and connected to the fuel reservoir by means of $\frac{1}{8}$-in. ID neoprene tubing. The plug in the bottom of the float chamber is drilled and a piece of $\frac{1}{8}$-in. copper tubing soldered in the plug. The copper tubing is joined to the special horizontal jet by a piece of $\frac{1}{8}$-in. ID neoprene tubing. Drainage is obtained by disconnecting the tubing from the end of the special jet. Care should be taken to see that the inside diameter of all tubes and fittings is larger than that of the horizontal jet, so that there is no restriction to the flow of fuel. This particularly applies to the hole through the valve seat of the float bowl. If the valve sticks in the open position, it is probably due to a taper on the point where it rests on its retainer washer. In this case the taper should be turned off, or a new needle made with a 90-deg shoulder. A dab of solder on the float tends to keep it from rotating and changing the fuel level.

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[4] Late models of the detonation meter (available from the Phillips Petroleum Co., Chemical Products Dept., Bartlesville, Okla.) are equipped with this switch; and the switch itself may be purchased from the Phillips Co. for installation in earlier models.

[5] Refinements of this system may be used, such as separately mounting the small float bowl and moving it up and down at a constant rate by means of electric or clock mechanisms.

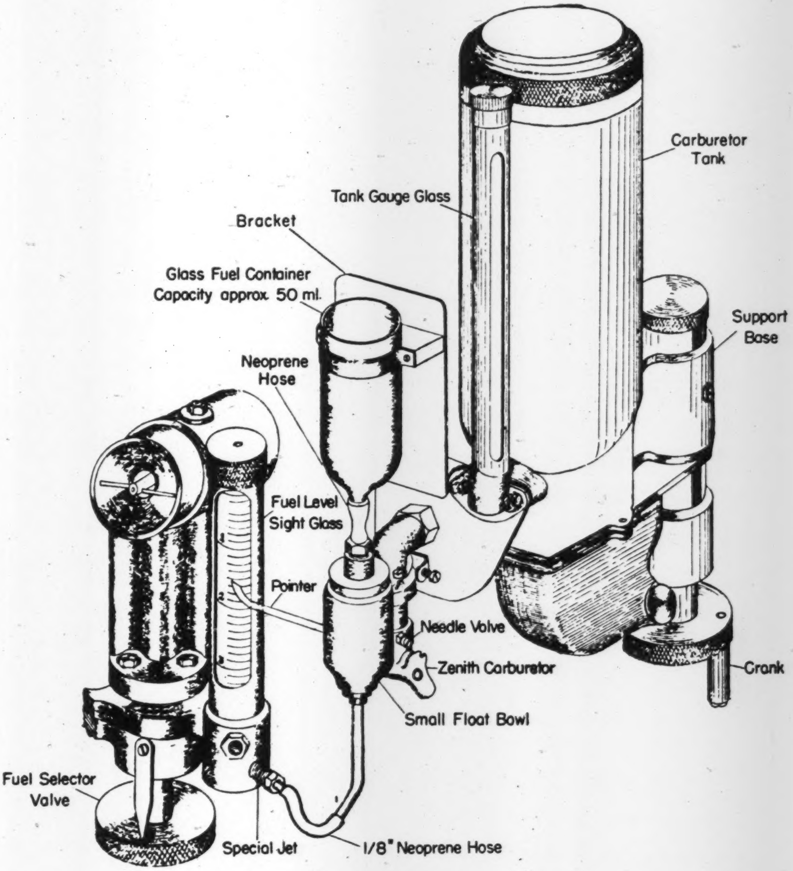
Carburetor Tank
Tank Gauge Glass
Bracket
Glass Fuel Container Capacity approx. 50 ml.
Support Base
Neoprene Hose
Fuel Level Sight Glass
Pointer
Needle Valve
Zenith Carburetor
Crank
Small Float Bowl
Fuel Selector Valve
Special Jet
1/8" Neoprene Hose

FIG. 1.—Small Float Bowl Fuel System.

(*e*) *Pointer.*—A pointer fitted to one hole in the boss of the choke control of the Zenith carburetor and extended to the graduated sight glass (which has been by-passed), provides a convenient float-level reference or datum. The pointer is made of $\frac{1}{8}$-in. copper tubing, flattened at one end, and the other end joined to the carburetor by a $\frac{1}{8}$-in. compression fitting. The pipe thread of the compression fitting is turned off for a press fit in the hole made for the choke control. The pointer should register zero on the sight gage when the position of the small float bowl just allows fuel to overflow the top of the vertical jet of the carburetor.

(*f*) *Crank.*—A crank is fitted to the adjusting screw of the right-hand fuel tank. Raising and lowering of the fuel tank is made much easier by turning the carburetor support post 90 deg from its present position and redrilling the body sleeve to insert the adjusting screw lift pin. This allows the weight of the fuel tank to bear on a solid surface instead of a slotted one.

Procedure

5. (*a*) *Preparation for Tests.*—Previous to starting tests on small samples, adjust the detonation meter to obtain a spread of 15 to 20 knockmeter divisions per octane number at the 85-octane number level. It may be necessary to readjust the meter to obtain sufficient spread or stability at very high or low octane levels, especially by the Research method. For this operation the regular fuel tanks are used with the short-time-constant switch of the meter in the slow-response position. All tests on fuel in the large tanks are made at the fuel level for maximum knock. Change the short-time-constant switch to a faster response up to the point where the knockmeter does not fluctuate more than ±3 divisions when running on one of the reference fuels used to obtain the spread. Fill the small reservoir with reference fuel similar to one in the large fuel tanks, turn selector valve to this fuel and, starting with the float bowl in a low position, slowly and continuously raise its level by means of the crank until it passes through the maximum-knock air–fuel ratio. Note the maximum knockmeter reading. This should be the same, approximately, as the reading obtained on the same fuel in the large tank, using the standard procedure. If not, either the rate of turning of the crank is too fast or further adjustment of the six-position short-time-constant switch is necessary.

(*b*) *Small-Sample Tests.*—Run the engine on *iso*octane from one of the large tanks. Pour some of the small sample into the fuel reservoir. If there is no previous guide as to its approximate octane number, set the cylinder micrometer at a value equivalent to about 80-octane number. Otherwise, set at the equivalent of the estimated octane number. With the small float bowl at a level which, from past experience, has been found to be close to that of maximum knock for most fuels, run on the sample momentarily and listen to the knock. If the compression ratio is such that it obviously does not give a knock intensity somewhere near standard for the method, switch to the *iso*octane in the large tank. Make a blend of reference fuel more nearly approaching the knock characteristics of the small sample. Place this in one of the large tanks, and run the engine on it while making the preliminary corrections to the compression ratio. The purpose of this blend is to avoid extreme variations in knock when changing to the small sample. Operate the engine on the small sample again and make the final adjustment of the compression ratio to give a knockmeter reading somewhere between 30 and 60 divisions. Starting from a lower (lean) level, slowly and continuously turn the small crank until the level passes through its maximum knock position; at the same time observe the maximum knockmeter reading. There is generally sufficient fuel to make three runs through the maximum knock level. Record these readings. If the first one differs materially from the other two, discard the first reading.

Switch to the large fuel tank and drain the sample. Using the cylinder micrometer reading as a guide, prepare bracketing reference-fuel blends differing by approximately two octane numbers. These must be run in the small float bowl at the same compression ratio used for the sample, varying the float level in a manner similar to that for the sample. However, as the amount of the reference fuel blends is not limited, the engine must be run on the blends long enough to clear out the manifold and to reach equilibrium conditions. This is particularly important if a sample containing tetraethyllead is of such a nature as to adhere to the manifold walls.

To avoid undue delay between testing the sample and the bracketing reference fuels, it is well to preblend a series of fuels differing by two octane numbers, or for the operator to obtain assistance in the blending operation.

Calculation and Report

6. Average the knockmeter readings obtained for the sample and for each of the reference-fuel blends, and interpolate the octane number in the usual manner. Results should be reported as "Small-Sample Octane Numbers."

Reproducibility

7. Tests on a number of samples of varying composition indicate that the reproducibility between different engines and laboratories is not quite as good as that for ASTM Methods D 357 and D 908.

PIPET FUEL SYSTEM

Apparatus

8. The fuel system, modified in accordance with Fig. 2, shall conform to the following requirements:

(*a*) *Jets.*—A special horizontal jet (as shown in Fig. 3), drill size 0.027 in. for the Motor method, and drill size 0.031 in. for the Research method. These sizes are approximate, and minor adjustments may be necessary (as in ASTM Methods D 357 and D 908) for individual engines, to bring the majority of maximum-knock float levels within 0.8 to 1.8 in. from the top of the vertical jet. The horizontal jet plug and the horizontal jet which serve the right-hand bowl of the carburetor, are removed. These are replaced by the special jet.

(*b*) *Pipet or Fuel Reservoir.*—The fuel reservoir is made of a glass tube with a capacity of 50 ml or more, and a minimum diameter of 25 mm. A convenient fuel reservoir can be made from a filter-tube funnel which has an inside diameter of 32 mm and an approximate capacity of 65 ml. To aid in measuring sample volume and for reference marks when determining fuel level for maximum knock, lines are etched and labeled on the glass tube to indicate sample volume at different fuel levels (Fig. 2). The glass tube is strapped to a brass mounting bracket (Fig. 2) which in turn is attached to the right-hand fuel tank of the carburetor where the tank-gage glass is bolted to the fuel tank. A piece of $\frac{1}{8}$-in. ID neoprene tubing is used to connect the outlet of the glass tube reservoir with the inlet of the special horizontal jet. Drainage is obtained by disconnecting the tubing from the end of the special jet.

(*c*) *Bracket.*—The bracket for mounting the glass fuel reservoir is shown in Fig. 3. Three holes are drilled in the top section of the bracket, such that the two outside holes correspond with the holes in the carburetor fuel-tank body. Standard fuel-tank-gage gaskets are placed on each side of the bracket before attaching the fuel-tank sight-glass gage. In this manner the modified fuel system may be left on the carburetor permanently, without interference with normal operation. Two holes are drilled in the center section of the bracket for bolting on a block of $\frac{1}{4}$-in. phenolic plastic. The glass reservoir is held in place by a thin strip of sheet metal fastened to the plastic block (Fig. 2). A thin band of neoprene gasket material between the sheet metal strip and the glass funnel will help hold the fuel reservoir more firmly in place.

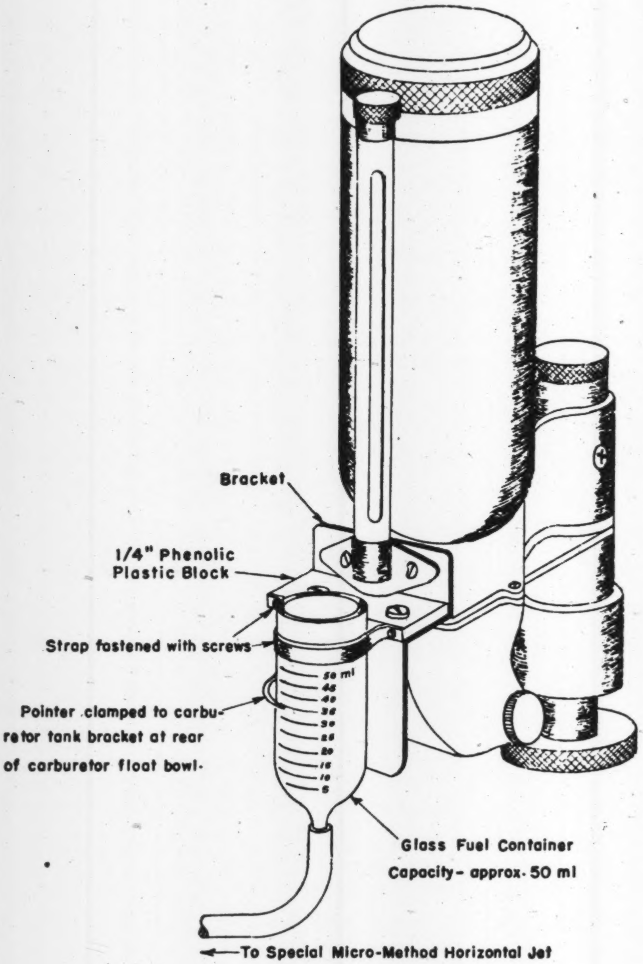
Bracket
1/4" Phenolic Plastic Block
Strap fastened with screws
Pointer clamped to carburetor tank bracket at rear of carburetor float bowl.
50 ml
45
40
35
30
25
20
15
10
5
Glass Fuel Container Capacity- approx. 50 ml
To Special Micro-Method Horizontal Jet

FIG. 2.—Pipet Fuel System.

(*d*) *Pointer.*—A drawing of the pointer assembly is shown in Fig. 4. The pointer is made of $\frac{1}{8}$-in. OD copper tubing which has been hooked, flattened and pointed at one end. The pointer is fastened to the carburetor tank bracket by means of a brass clamp. The brass clamp is held to the carburetor tank bracket by a $\frac{1}{4}$-in. Allen set screw at a point midway between the center and right-hand carburetors. A hole is drilled at the rear of the brass clamp for the pointer, which is locked in position at any desired height by a small knurled thumb screw.

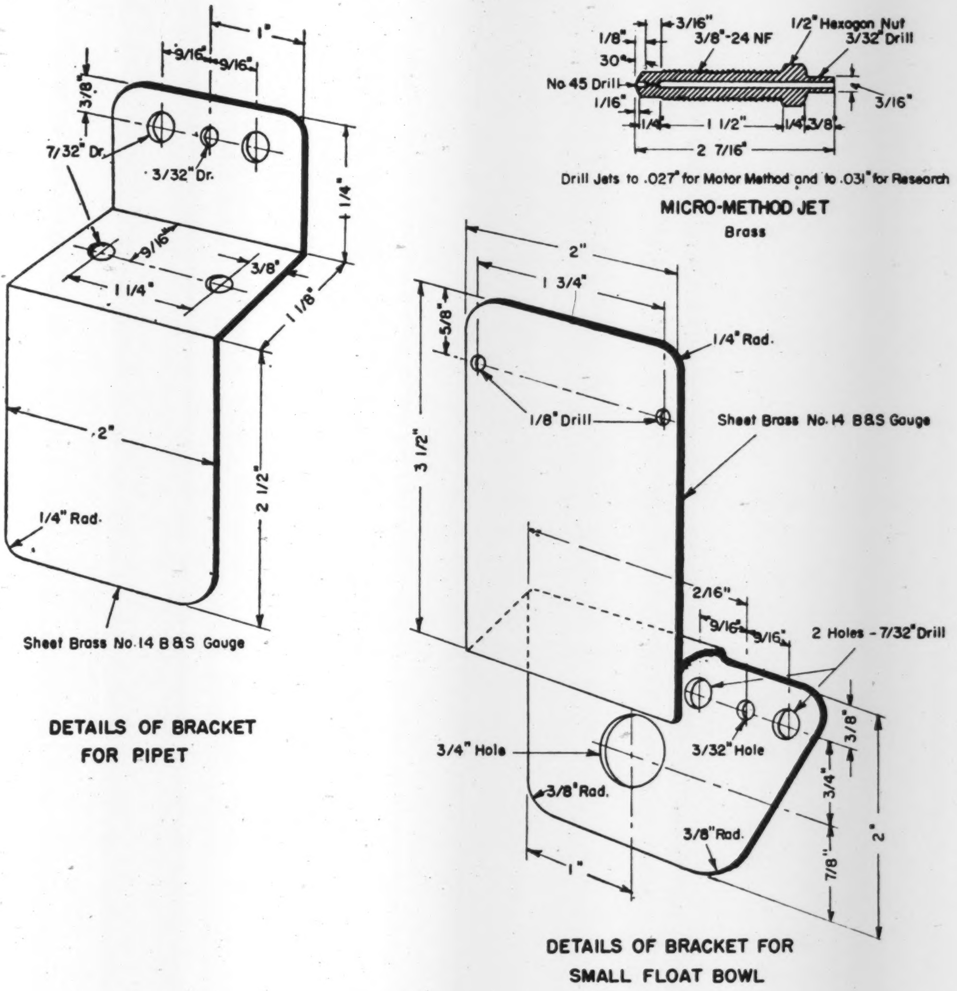
1/8"
3/16"
3/8"-24 NF
1/2" Hexagon Nut
3/32" Drill
30°
No. 45 Drill
1/16"
1/4
1 1/2"
1/4
3/8
3/16"
2 7/16"
Drill Jets to .027" for Motor Method and to .031" for Research
MICRO-METHOD JET
Brass
1"
9/16"
9/16"
3/8"
7/32" Dr.
3/32" Dr.
1/4"
9/16"
3/8"
1 1/4"
1 1/8"
2"
2 1/2"
1/4" Rad.
Sheet Brass No. 14 B&S Gauge
DETAILS OF BRACKET FOR PIPET
2"
1 3/4"
5/8"
1/4" Rad.
1/8" Drill
Sheet Brass No. 14 B&S Gauge
3 1/2"
2/16"
9/16"
9/16"
2 Holes - 7/32" Drill
3/4" Hole
3/32" Hole
3/8" Rad.
3/8" Rad.
3/8"
3/4"
7/8"
2"
1"
DETAILS OF BRACKET FOR SMALL FLOAT BOWL

FIG. 3.—Details of Brackets and Micro Jet.

Procedure

9. (*a*) *Preparation for Tests.*—Previous to starting tests on small samples, adjust the detonation meter to obtain a spread of 15 to 20 knockmeter divisions per octane number at the 85-octane number level. It may be necessary to readjust the meter to obtain sufficient spread or stability at very high or low octane levels, especially by the Research method. For this operation the regular fuel tanks are used with the short-time-constant switch of the meter in the slow-response position. All tests on fuel in

the large tanks are made at the fuel level for maximum knock. Change the short-time-constant switch to a faster response up to the point where the knockmeter does not fluctuate more than ±3 divisions when running on one of the reference fuels used to obtain the spread. Fill the small glass tube reservoir with reference fuel similar to one in the large fuel tanks, turn selector valve to this fuel and, with a given setting of the adjusting screw, note the knockmeter reading at which maximum knock occurs as the fuel is consumed by the engine. The reading at maximum-knock air–fuel ratio should be the same, approximately, as the reading obtained on the same fuel in the large tank, using the standard procedure. If not, rerun the test with an adjusting-screw setting which allows maximum knock to occur when the fuel level in the glass tube is near the bottom of the tube. If difficulty is still encountered with matching of the maximum knockmeter readings, further adjustment of the six-position short-time-constant switch is necessary.

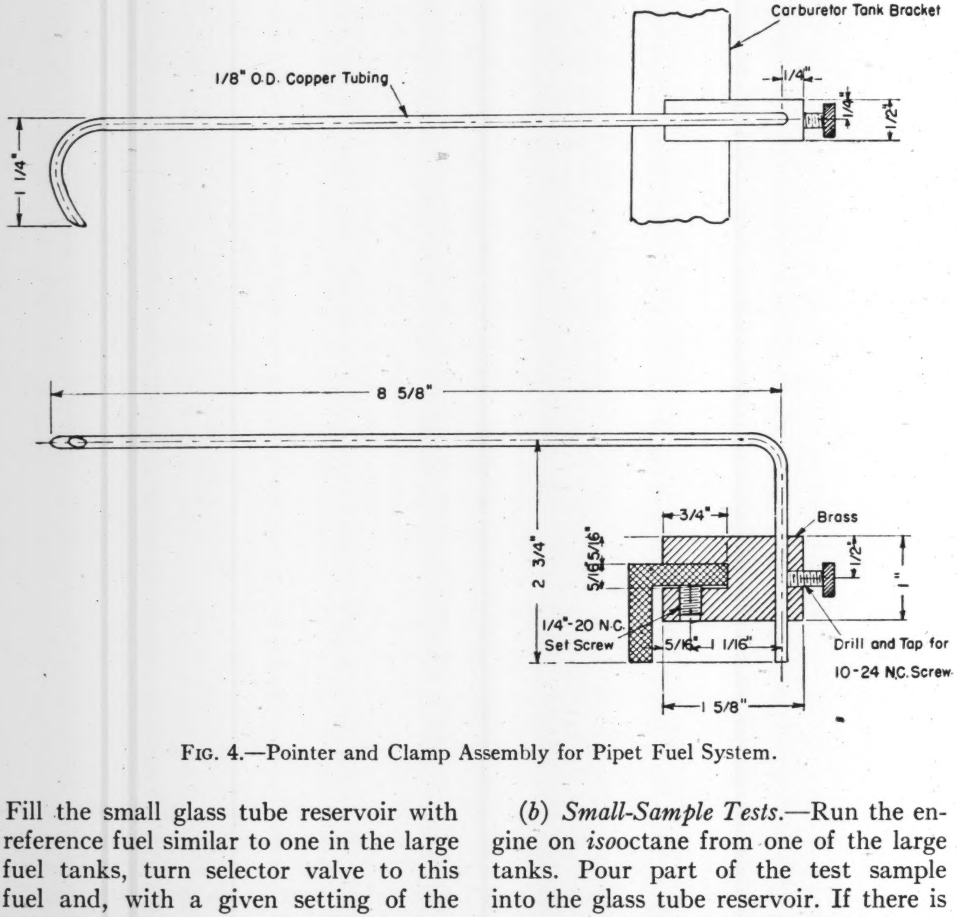

FIG. 4.—Pointer and Clamp Assembly for Pipet Fuel System.

(*b*) *Small-Sample Tests.*—Run the engine on *iso*octane from one of the large tanks. Pour part of the test sample into the glass tube reservoir. If there is no previous guide as to its approximate octane number, set the cylinder micrometer at a value equivalent to about 80-octane number. Otherwise, set at the equivalent of the estimated octane number. Adjust the height of the glass tube reservoir with the adjusting screw so that, from past experience, the fuel level will be close to or slightly above that of maximum knock for most fuels. Run on the sample momentarily and listen to the knock. If the compression ratio is such that it obviously does not give a knock intensity somewhere near standard for the method, switch to *iso*octane in the large tank. Make a blend of reference fuel more nearly approaching the knock characteristics of the small sample. Place this in one of the large tanks, and run the engine on it while making the preliminary corrections to the compression ratio. The purpose of this blend is to avoid extreme variations in knock when changing to the small sample. Operate the engine on the small sample again and make the final adjustment of the compression ratio to give a knockmeter reading somewhere between 40 and 60 divisions. By this time the position of the fuel level for maximum knock with relation to the pointer is fairly well established. If desired, the position of the pointer can be reset (by means of the locking screw in the clamp) at the fuel level for maximum knock, and more than one pass can generally be made through the maximum-knock range by either adding additional sample or by raising the small glass tube so that the fuel level is above that of the pointer. Record these readings.

Switch to the large fuel tank and drain the sample. Using the cylinder micrometer reading as a guide, prepare bracketing reference-fuel blends differing by two octane numbers. These must be run with the glass tube reservoir at the same compression ratio used for the sample, determining the maximum knock in a manner similar to the sample. However, as the amount of the reference-fuel blends is not limited, the engine must be run on the blends long enough to clear out the manifold and to reach equilibrium conditions. This is particularly important if a sample containing tetraethyllead is of such a nature as to adhere to the walls of the manifold.

To avoid undue delay between testing the sample and the bracketing reference fuels, it is well to preblend a series of fuels differing by two octane numbers, or for the operator to obtain assistance in the blending operation.

Calculation and Report

10. Average the knockmeter readings obtained for the sample and for each of the reference-fuel blends, and interpolate the octane number in the usual manner. Results should be reported as "Small-Sample Octane Numbers."

Reproducibility

11. Tests on a number of samples of varying composition indicate that the reproducibility between different engines and laboratories is not quite as good as that for ASTM Methods D 357 and D 908.

APPENDIX VII

PROPOSED METHOD OF TEST FOR MOTOR AND RESEARCH OCTANE NUMBERS OF SMALL SAMPLES BY MULTI-PIPET SYSTEM[1]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

Scope

1. This method describes a "small sample" test for determining the knock characteristics, in terms of either Motor octane numbers or Research octane numbers, of fuels for use in spark-ignition engines. The principal difference between this method and the corresponding ASTM Method D 357, Standard Method of Test for Knock Characteristics of Motor Fuels by the Motor Method,[2] and ASTM Method D 908, Standard Method of Test for Knock Characteristics of Motor Fuels by the Research Method,[2] is that this method can be used on small samples. Not over 50 ml of the fuel is required, and if some knowledge of the probable octane number is available, the quantity can be reduced to approximately 25 ml.

Outline of Method

2. In the small-sample method, the principle of the falling fuel level is used to obtain the maximum knock intensity (at the correct compression ratio) for both known or reference fuels, and for unknown fuels. Due to the short duration of the maximum intensity peak, a quick-response meter is required. By bracketing the meter reading of the unknown fuel between two known reference fuels, the octane number is calculated.

Apparatus

3. In addition to the ASTM-CFR engine (Motor or Research) and necessary accessories as described in detail in the respective ASTM Methods D 357 and D 908, the following changes and additions in equipment are required:

(*a*) *Detonation Meter, Model 501, and Detonation Pickup, Type D-1*,[3] are used instead of a bouncing pin. The detonation meter should be equipped with a six-position variable time-constant switch.[4]

(*b*) *Small-Sample Reservoir Assembly*, as shown in Fig. 1, consisting of four identical bowls, each of about 40-ml capacity. These are manifolded through stopcocks, capillary glass tubing, and flexible tubing to the carburetor jet. Plastic or glass covers are provided to keep out dust and reduce evaporation losses. The bowls are made in units of two for flexibility, standardization, and ease of replacement. A removable plug is placed at the opposite end of the manifold to permit cleaning of the capillary bore in the event it might become plugged with foreign matter.

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[1] This proposed method is under the jurisdiction of the ASTM Committee D-2 on Petroleum Products and Lubricants. Published as information, June, 1951.

[2] 1949 Book of ASTM Standards, Part 5.

[3] This apparatus, including variable time-constant switch, may be obtained from the Phillips Petroleum Co., Chemical Products Dept., Bartlesville, Okla.

[4] This equipment (excepting the switch) is described in the 1950 Appendix to the ASTM Manual of Engine Test Methods for Rating Fuels, issued January, 1950

(*c*) *Gasoline-Insoluble Grease,* to lubricate the glass stopcocks. A suitable lubricant may be prepared as follows:

	Parts by Weight
Anhydrous glycerin	25
Dextrin	7
d-Mannitol	35

Mix, heat to 220 F, and cool. Since this lubricant is water-soluble, it may be removed by means of water.

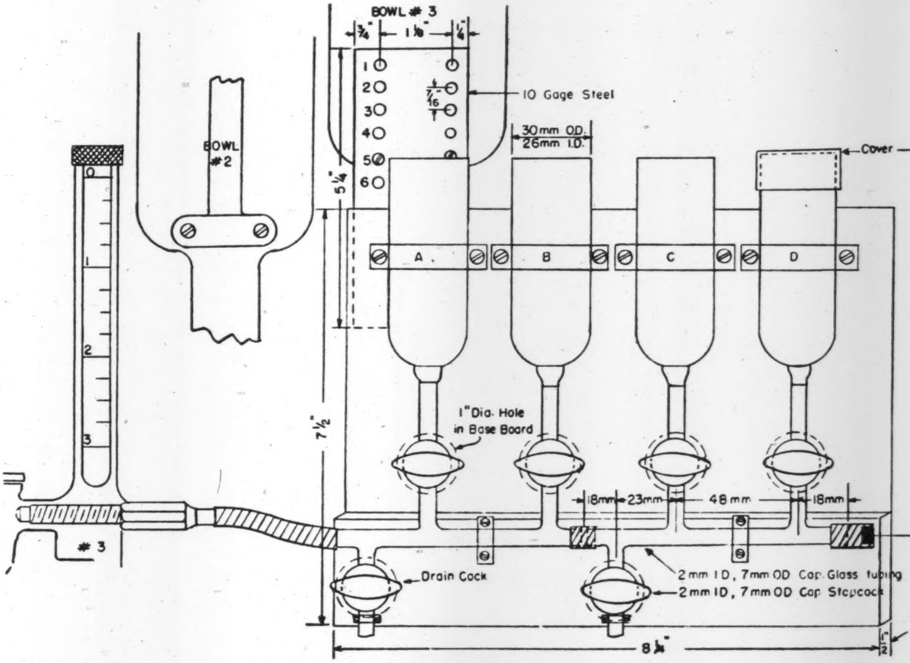

BOWL # 3
3/4"
1 1/8"
1/4"
10
20
30
40
50
60
1/4
1/16
10 Gage Steel
30mm OD.
26mm I.D.
Cover
BOWL #2
5 1/4"
7 1/2"
A
B
C
D
80mm
73mm
1" Dia. Hole in Base Board
18mm
23mm
48 mm
18mm
# 3
Drain Cock
2 mm I D, 7mm OD Cap. Glass tubing
2mm ID, 7mm OD Cap. Stopcock
8 1/2"
1/2"

FIG. 1.—Small-Sample Reservoir Assembly.

(*d*) *Special Carburetor Jet,* as shown in Fig. 2. For the Research method, the orifice should be a No. 67 drill size, and for the Motor method, a No. 71 drill size. This jet can be made from brass or any other suitable metal.

(*e*) *One-quarter-Inch Flexible Tubing* made of Saran or similar gasoline-insoluble material, for making connections.

Reference Materials

4. Reference Materials are the same as those listed under ASTM Motor Method D 357 and ASTM Research Method D 908.

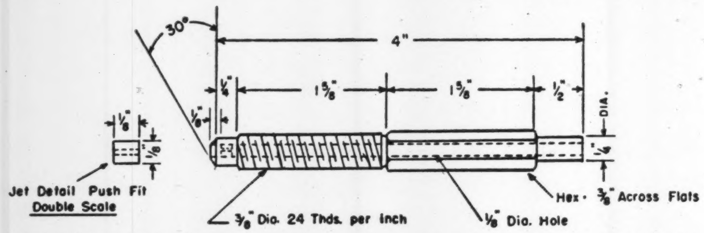
Operating Conditions

5. Operating conditions are the same as those given for ASTM Motor Method D 357 or for ASTM Research Method D 908.

Assembly of Apparatus

6. (*a*) Insert the special Motor or Research carburetor jet recommended in place of the No. 3 bowl's regular jet and plug. The No. 3 bowl should now be set midway in its screw adjustment. In order to mount the small-sample reservoir assembly on the No. 3 bowl, it is necessary to remove the fuel-tank sight-glass assembly. In case it is desired to use the No. 3 bowl for ASTM Method D 357 or D 908 as well as for the Small-Sample method, a bushing can be inserted to offset the sight-glass assembly so that it may still be used with

the small-sample assembly in place. In either case, the small-sample assembly should be attached to the No. 3 bowl, using the same holes provided for attachment of the fuel-tank sight-glass assembly. The small-sample reservoir assembly is provided with a mounting bracket which has a series of holes by which it is possible to extend the fuel-level range normally provided by the carburetor-tank adjusting screw. Normally, position 5 will permit the maximum-knock fuel level to come within the 0.8 to 1.8-in. acceptable limits, as measured by projecting the fuel level across to the now inactive calibrated fuel-level gage.



NOTE.—Orfice should be No. 67 drill size for the Research method and No. 71 drill size for the Motor method.

FIG. 2.—Carburetor Jet.

(*b*) If it is desired to obtain the octane numbers of fuels having very high or low viscosities, difficulty may be encountered in complying with the 0.8 to 1.8-in. range allowed. These fuels may be tested by making necessary changes in the jet size, such as is done in ASTM Method D 357 or D 908. If, however, for the sake of expediency, less accuracy may be tolerated, these fuels may be run using the normal jet. This is accomplished by altering the height of the small-sample reservoir assembly through the use of the extra holes provided in the mounting bracket. In this case, of course, the fuel level may be outside the specified range.

Procedure

7. (*a*) *Starting and Stopping Engine.*—Follow the procedure described in ASTM Motor Method D 357 and ASTM Research Method D 908.

(*b*) *Preparation for Test.*—With the fuel-selector valve turned to the No. 1 bowl and with an unleaded gasoline (such as 90-octane alkylate) in the No. 1 bowl, bring the engine to equilibrium in compliance with the standard test conditions. The detonation meter must be "on" for a sufficient time to provide for thermal equilibrium and no drift. This normally takes about 30 min. If necessary, the zero adjustment should be made in accordance with the method given in the 1950 Appendix to the ASTM Manual of Engine Test Methods for Rating Fuels. The compression ratio on the motor is now adjusted so that the micrometer setting will correspond to that shown for 85-octane number in the standard knock-intensity guide tables.

(*c*) *Calibration of the Motor.*—Place an 84-octane number reference fuel in small-sample reservoir A and an 86-octane number reference fuel in small-sample reservoir B (see Fig. 1). Open the stopcock of the A reservoir, and turn the carburetor fuel-selector valve to position No. 3, which places the small-sample reservoir assembly into the system. By alternately running on the reference fuels in reservoirs A and B, the detonation meter is now adjusted for meter reading and spread according to the 1950 Appendix of the ASTM

Manual, except for the following deviations:

(*1*) The small-sample reservoir system is used.

(*2*) No adjustment is made on the bowl height to obtain the maximum knock air–fuel ratio. Instead, the fuel is allowed to flow into the carburetor without any adjustment of the glass reservoir. As the fuel is consumed and the level falls, at some point the detonation will reach a maximum, which should occur within the previously specified limits of 0.8 to 1.8 in. as sighted on the carburetor fuel-level gage. Corrective adjustment of the carburetor-tank adjusting screw is necessary should the maximum knock level occur outside of these limits.

(*3*) The variable time-constant switch is set to give maximum speed for the required needle stability. Knockmeter fluctuations of 3 or 4 divisions are not considered excessive, and can usually be obtained on position 2 or 3, depending on the condition of the motor.

(*4*) The meter reading and spread dials are adjusted to give 15 to 20 divisions per octane number, centering on the 50-division line.

(*5*) In any calibration work, and also when testing an unknown fuel, the change from one fuel to another is made by changing the carburetor-bowl fuel-selector valve to position 1 (the 90-octane stand-by fuel) (Note 1), closing the glass stopcock of the glass reservoir just run, opening the glass stopcock of the next reservoir to be run, and turning the carburetor-bowl fuel-selector valve to position 3. This fuel change is usually made in 5 sec or less.

NOTE 1.—When the engine has been running for several minutes on the stand-by fuel (position 1) and is switched to the glass reservoir system, the first run will usually give a somewhat low meter reading. For this reason, the first small-sample run should be made on an appropriate reference fuel; and although the reading is recorded, it is omitted in averaging the final results. It is desirable to record the numerical sequence of each small-sample operation, so that if the engine drifts, the appropriate meter readings can be selected for interpolation.

(*6*) At no time during either calibration or testing of unknown samples should the motor be allowed to run out of fuel, for this will upset the thermal equilibrium and result in a delay in re-establishing proper operating conditions. Whenever the engine is not running on fuel from a glass reservoir, it should run on the 90-octane fuel in bowl No. 1.

(*d*) *Checking Test Conditions by Standardization Fuel.*—Test conditions should be checked by the procedure given under Motor Method D 357 or Research Method D 908, as amended by the 1950 Appendix to the ASTM Manual, except that the small-sample assembly is used.

(*e*) *Determination of Knock Rating of Unknown Fuel.*—With the motor operating on the 90-octane number fuel in bowl No. 1, pour approximately 35 ml of unknown sample into reservoir A. An estimation of the probable knock rating of the sample, based on previous history or operator experience, is highly desirable, as in Methods D 357 and D 908, to properly make the preliminary compression-ratio adjustment. For example, if it is estimated that the sample has an 80-octane number, pour a known 80-octane number reference fuel into reservoir B, and open the corresponding stopcock. Then set the compression ratio to conform to the guide-table micrometer setting. Switch the fuel selector valve to position 3 (the small-sample assembly) and allow the engine to operate on this fuel. In view of the limited quantity of sample, the observed readings on the knockmeter will rise rapidly as the fuel–air ratio approaches the maximum knock point. The highest reading noted (Note 2) is recorded as the maximum knock point. Now rapidly refill reservoir B and recheck the maximum knock point. If these values are not within 3 to 5 units, a third check will be necessary. This maximum knock should occur at approximately the middle of the meter scale.

NOTE 2.—Take the maximum meter reading as the highest value that persists for at least 2 sec. Disregard an occasional high flick of the meter needle. If the sample volume is limited, it can be conserved by turning the selector valve to position 1 as soon as the meter reading starts to fall from the maximum.

Using the sequence described under Paragraph (*c*), item (*5*), allow sufficient test fuel to flow to determine whether the estimation is in the proper range. If it is in the proper range, proceed as before to determine the maximum knock point. Obtain a check knockmeter reading by refilling with the sample to about ½ to ¾ in. above maximum-knock fuel level. Place a second bracketing reference fuel in reservoir C, and make the rating in the same way. The two bracketing reference fuels should not differ by more than two octane numbers. If the sample volume permits, it is advisable to repeat the determination (Note 3).

NOTE 3.—In small-sample-method testing, no attempt should be made to run bracketing reference fuels in the regular ASTM carburetor.

If the unknown fuel is not in the anticipated octane number range, immediately switch back to position 1, reset the compression ratio to a better value, and repeat the determination previously described. An experienced operator will need only a few ml of fuel to ascertain the proper range.

In testing a series of fuels having somewhat similar octane numbers, it is advantageous to put a series of reference fuels in reservoirs B, C, and D, differing from each other by two octane numbers and covering the expected range. It is also advantageous to have a wide range of made-up reference fuels at hand in pint bottles.

Calculation and Report

8. Average the knockmeter readings obtained for the sample and for each of the reference fuel blends, and find the Motor or Research octane number by interpolation from the averages so obtained. Report the octane number (Small-Sample Method) to the nearest 0.1.

Reproducibility

9. The accuracy of the Small-Sample methods, either Research or Motor, is not quite as good as the corresponding ASTM Methods D 357 and D 908. The time required to rate a sample is approximately 25 per cent of that required for the corresponding ASTM methods.

REPORT OF COMMITTEE D-3

ON

GASEOUS FUELS*

Four new methods covering sampling, determination of water vapor content, and analysis of gaseous fuels were completed and approved by Committee D-3 during the year. All were accepted as tentative.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1950 Annual Meeting, Committee D-3 presented to the Society through the Administrative Committee on Standards the following recommendations:

Tentative Method of:

Test for Analysis of Natural Gases by the Volumetric Chemical Method (D 1136 - 50 T),

Test for Analysis of Natural Gases and Related Types of Gaseous Mixtures by the Mass Spectrometer (D 1137 - 50 T),

Test for Water Vapor Content of Gaseous Fuels by Measurement of Dew Point Temperature (D 1142 - 50 T), and

Sampling Natural Gas (D 1145 - 50 T).

The first two methods were accepted by the Standards Committee on September 26, 1950; D 1142 - 50 T was accepted on December 14, 1950, and D 1145 - 50 T on March 2, 1951. The methods appear in the 1950 Supplement to Book of ASTM Standards, Part 5, and also in the 1951 Compilation of ASTM Standards on Gaseous Fuels.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Collection of Gaseous Samples (F. E. Vandaveer, chairman).—The new Tentative Method for Sampling Natural Gas was prepared by this subcommittee. Two other methods covering sampling of manufactured gas and of liquefied petroleum gases are in preparation.

Subcommittee IV on Determination of Specific Gravity and Density of Gaseous Fuels (E. F. Schmidt, chairman).—Since the Tentative Methods of Test for Specific Gravity of Gaseous Fuels were published in December, 1949, a number of suggestions for revision have been received, mostly of an editorial nature. They will be reviewed by the subcommittee. Barring any major revisions of the tentative methods, which are not now evident, the subcommittee will recommend that the methods as modified be adopted as standard.

Subcommittee VII on Complete Analysis of Chemical Composition of Gaseous Fuels (Martin Shepherd, chairman).—The two new tentative methods of analysis were prepared by this subcommittee. Work is also in progress on additional test methods including the preparation of samples for analysis by cooperating laboratories.

This report has been submitted to letter ballot of the committee, which consists of 37 members; 30 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

A. W. GAUGER,
Chairman.

K. R. KNAPP,
Secretary.

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* Presented at the Fifty-fourth Annual Meeting of the Society, June 18-22, 1951.

REPORT OF COMMITTEE D-4
ON
ROAD AND PAVING MATERIALS*

Committee D-4 on Road and Paving Materials has held two meetings during the past year: in Atlantic City, N. J., on June 29, 1950, and in Cincinnati, Ohio, on March 8, 1951.

Recommendations Accepted by the Administrative Committee on Standards

Subsequent to the 1950 Annual Meeting, Committee D-4 presented to the Society, through the Administrative Committee on Standards, recommendations for publication of the following:

Tentative Specifications for:

Crushed Stone, Crushed Slag, and Gravel for Single Bituminous Surface Treatment (D 1139 - 50 T), and

Tentative Method of Test for:

Resistance to Plastic Flow of Fine-Aggregate Bituminous Mixtures (D 1138 - 50 T).

These tentative specifications and tentative method of test were accepted by the Administrative Committee on Standards on September 26, 1950, and the new tentatives appear in the 1950 Supplement to ASTM Book of Standards, Part 3.

Tentative Revision of Standard

Standard Specifications for Emulsified Asphalt (**D 977 - 49**).[1]—Committee D-4 recommends the following tentative revisions of the Standard Specifications D 977 to provide requirements for an additional grade of emulsified asphalt, which has rapid-setting high-viscosity properties and is suitable for surface treatment:

Section 1.—Change the description of the first type to read as follows, by the addition of the italicized words:

> *Type RS-1.*—Emulsified asphalt (quick-setting, *low-viscosity*) for penetration and surface treatment.

Insert the following new sentence as the second type of emulsified asphalt:

> *Type RS-2.*—Emulsified asphalt (quick-setting, high viscosity) for surface treatment.

Table 1.—Under the heading "Quick-Setting," insert requirements for type "RS-2," as follows:

	Min.	Max.
Viscosity, Saybolt Furol, 60 ml at 50 C (122 F), sec.	75	400
Residue by distillation, per cent.	63	68
Settlement, 5 days	...	3
Demulsibility, 35 ml of 0.02 *N* $CaCl_2$, per cent	60	...
Sieve Test	...	0.10

Sections 3 and 4.—Change the last sentence to read as follows by the addition of the words in italics:

> For type RS-1 *or type RS-2*, containers shall be glass or black iron.

Standard Definitions of Terms Relating to Materials for Roads and Pavements (**D 8 - 49**).[1]—The committee recommends that the tentative revision of this standard, issued June, 1949, be amended by deleting the definition of "viscosity" and reference to that definition in the introduction. This definition of "viscosity," as well as that appearing in the standard, has received much criticism; furthermore, viscosity is a fundamental property of matter which has received consideration by Committee E-1 on Methods of Testing, and a definition of "viscosity" is incorporated in the definition of "Simple Liquid" of the Standard Definitions of Terms Relating to Rheological Properties of Matter (E 24 – 42). In view of the existence of this latter definition, Committee D-4 believes that it is not necessary or desirable to retain the inaccurate and questionable definitions appearing in Standard Definitions D 8 – 49 and in the tentative revision issued in June, 1949.

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* Presented at the Fifty-fourth Annual Meeting of the Society, June 18-22, 1951.

[1] 1949 Book of ASTM Standards, Part 3.

REVISIONS OF STANDARD, IMMEDIATE ADOPTION

Standard Definitions of Terms Relating to Materials for Roads and Pavements (**D 8 – 49**).[1]—Committee D-4 recommends that these standard definitions be revised by the withdrawal of the definitions for "Artificial Asphalt," "Blown Petroleums," and "Viscosity," and requests the necessary nine-tenths affirmative vote at the Annual Meeting in order that the recommendation may be referred to letter ballot of the Society for immediate adoption.

In the standard definitions, "artificial asphalt" is not defined; the statement following the term is merely a recommendation that use of the term be discontinued. The definition of "blown petroleums" is faulty because it limits application of the term to products from liquid native bitumens. Moreover, it is believed that definition of the term, if necessary, should more properly be the responsibility of Committee D-2 than of Committee D-4. Reasons for recommending withdrawal of the definition for "viscosity" have already been stated above.

ADOPTION OF TENTATIVES AS STANDARD

Committee D-4 recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard, without revision:

Tentative Specifications for Bituminous Mixing Plant Requirements (**D 995 – 49 T**),[1]

Tentative Methods of Sampling Bituminous Paving Mixtures (**D 979 – 49 T**),[1] and

Tentative Recommended Practice for Bituminous Mixing Plant Inspection (**D 290 – 49 T**).[1]

ADOPTION OF TENTATIVE REVISIONS AS STANDARD

Standard Method of Test for Abrasion of Coarse Aggregate by Use of the Los Angeles Machine (**C 131 – 47**).[1]—Committee D-4 joins with Committee C-9 on Concrete and Concrete Aggregates in recommending that the tentative revisions of this method, issued in June, 1949, be approved for reference to letter ballot of the Society for adoption as standard.

TENTATIVES CONTINUED WITHOUT REVISION

The subcommittees of Committee D-4 and the joint subcommittee of Committees D-4 and D-18 have reviewed existing tentative specifications and methods of test which have been published by the Society for two years or more without revision, and Committee D-4 recommends that the following specifications and methods be continued as tentative, without revision:

Tentative Specifications for:

Materials for Stabilized Base Course (D 556 – 40 T) and Materials for Stabilized Surface Course (D 557 – 40 T).—The joint subcommittee of Committees D-4 and D-18, in cooperation with a committee of the American

Association of State Highway Officials, is continuing its efforts to combine these two specifications and to prepare simplified and more direct requirements for suitable materials.

Asphalt Cement for Use in Pavement Construction (D 946 - 47 T).—These specifications should be retained as tentative while Subcommittee C-1 develops additional requirements to secure improved control of the properties and uniformity of asphalt cements.

Hot-Mixed, Hot-Laid Asphaltic Concrete for Base and Surface Courses (D 947 - 49 T).—The subcommittee is considering a revision of this specification involving the addition of two sizes of aggregate and the rewording of Section 5 on composition of mixtures.

Asphaltic Mixtures for Sheet Asphalt Pavements (D 978 - 49 T).—The subcommittee proposes to revise this specification by changing the wording of Section 5 on composition of mixtures.

Preformed Expansion Joint Filler for Concrete, Bituminous Type (D 994 - 48 T).—Subcommittee D-3 is considering several suggestions for changes in requirements and in the methods of testing, as well as a proposal to consolidate this specification with others for materials of similar use.

Fine Aggregate for Sheet Asphalt and Bituminous Concrete Pavements (D 1073 - 49 T).—Subcommittee C-10 is considering a proposal to revise this specification by adding a section on soundness of aggregate.

Tentative Methods of Test for:

Soft Particles in Coarse Aggregates (C 235 - 49 T).—This tentative method is under the joint jurisdiction of Committee D-4 and Committee C-9 on Concrete and Concrete Aggregates. Subcommittee B-12 is considering several suggestions for revision of requirements.

Bitumen (D 4 - 49 T).—Suggestions have been made to consider the influence of carbonates and ignition losses on the determination, and Subcommittee B-21 is conducting a cooperative program of tests which may lead to recommendations for revision of the tentative method.

Loss on Heating of Oil and Asphaltic Compounds (D 6 - 39 T). As mentioned in the 1950 Annual Report of Committee D-4, this tentative method has been in extensive use for many years, and should be retained until an improved procedure is adopted.

Moisture-Density Relations of Soils (D 698 - 42 T).—This tentative method is under the joint jurisdiction of Committee D-4 and Committee D-18 on Soils for Engineering Purposes. The latter committee is considering revision of this method.

Soil-Bituminous Mixtures (D 915 - 47 T).—As indicated in the 1950 Annual Report of Committee D-4, further study of this procedure is desirable in respect to correlation with construction control and the service performance of stabilized highway soils. A subcommittee of Committee D-18, which has joint jurisdiction with Committee D-4 over this method, proposes to assemble appropriate data on the applicability and utility of the tentative method under practical conditions.

Shear Strength of Flexible Road Surfaces, Subgrades, and Fills by the Burggraf Shear Apparatus (D 916 - 47 T).—The tentative method should be retained pending further development, as indicated in the 1950 Annual Report of Committee D-4.

Compressive Strength of Bituminous Mixtures (D 1074 - 49 T), and Effect of Water on Cohesion of Compacted Bituminous Mixtures (D 1075 - 49 T).—Subcommittee B-25 is conducting cooperative tests in a study of reproducibility of these methods and is cooperating with Subcommittee B-2 in further development and standardization of procedures, considering various types of bituminous mixtures.

Tentative Methods of Sampling:

Bituminous Materials (D 140 - 49 T).—Subcommittees of Committee D-4 and Committee D-8 on Bituminous Waterproofing and Roofing Materials are cooperating in preparation of an extensive revision of these tentative methods.

The recommendations appearing in this report have been approved by letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee B-1 on Asphalt Content (J. H. Swanberg, chairman) has prepared a new tentative method of test for the determination of residue of a specified penetration. The method involves distillation under vacuum, and the subcommittee has completed an extensive series of cooperative tests with 14 samples of SC-3 road oil, comparing the proposed method with two other methods published by the Society.

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[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee B-2 on Physical Tests for Compressed Bituminous Mixtures (L. F. Rader, chairman) and *Subcommittee B-25 on Effect of Water on Compressed Bituminous Mixtures* (C. A. Carpenter, chairman) are planning to cooperate in development of compressive strength tests for mixtures comprising liquid asphaltic materials and the effect of water on such compressed mixtures. Subcommittee B-2 has prepared a proposed method for determination of specific gravity of compressed bituminous mixtures and plans a cooperative investigation of methods for the preparation of test specimens to be subjected to various types of strength test. This subcommittee is also considering the development of additional procedures in a series of standardized strength tests, including triaxial compression tests and the Marshall test. Subcommittee B-25 is engaged in a thorough study of the reproducibility to be expected with Tentative Method of Test for Effect of Water on Cohesion of Compacted Bituminous Mixtures, D 1075 – 49 T.

Subcommittee B-3 on Distillation (H. P. Ferguson, chairman) has prepared a revision of the Standard Method of Test for Distillation of Tar Products Suitable for Road Treatment (D 20 – 30) and plans to conduct a series of cooperative tests to obtain data on the precision to be expected with this test. The proposed revision is largely editorial and is published for the information of interested members of the Society as Appendix II of this report.

Subcommittee B-5 on Softening Point (I. E. Manning, chairman) is continuing its efforts, in cooperation with Committee E-1, to develop a satisfactory method which will replace the two methods for Ring-and-Ball Softening Point now published by the Society.

Subcommittee B-7 on Viscosity and Float Tests (C. A. Benning, chairman) plans to conduct cooperative tests to obtain data on reproducibility of the Engler specific viscosity test, for which a proposed tentative method has been prepared.

Subcommittee B-14 on Specific Gravity of Fine and Coarse Aggregates (H. T. Williams, chairman) has been making a laboratory investigation of methods for the specific gravity of aggregates which will have direct significance in the control and design of paving mixtures.

Subcommittee B-16 on Setting Qualities of Bituminous Materials (A. B. Cornthwaite, chairman) has completed a series of cooperative tests showing reasonably good agreement in determination of setting time for the types of material included in the study. The subcommittee may be able to recommend a new tentative method based on this investigation.

Subcommittee B-17 on Emulsion Tests (R. R. Thurston, chairman) has been conducting cooperative tests to establish a procedure for determination of viscosity of emulsified asphalts at 122 F., as will be required in the proposed specifications for an RS-2 type of emulsified asphalt.

Subcommittee C-12 on Bituminous Surface Treatments (W. H. Mills, chairman) has made preliminary plans for the development of additional specifications and recommended practices applicable to surface treatment of highways.

Subcommittee D-3 on Expansion Joint Materials (D. O. Woolf, chairman) has prepared revisions of standard specifications for Preformed Expansion Joint Fillers for Concrete (Nonextruding and Resilient Types) and additional specifications for expansion joint materials, which are to be considered by the subcommittee before recommendations will be made for approval of Committee D-4.

A special subcommittee has been organized to study suggestions that requirements for the pycnometer specified in Standard Method of Test for Specific Gravity of Road Oils, Road Tars, Asphalt Cements, and Soft Tar Pitches (D 70 - 27) may be improved.

RESEARCH ACTIVITIES

The subcommittees of Committee D-4 have made considerable progress in study of the problems listed in the report presented at the 1949 Annual Meeting. This is evidenced by several recommendations made in this report and by the preceding summary of subcommittee activities. Of particular interest is the continued development of laboratory methods for the physical testing of compacted bituminous mixtures, and the study by Subcommittees B-2, B-25, and B-26 of methods proposed for the evaluation of bituminous mixtures and their performance under service conditions. Subcommittee B-26 has for several years been conducting cooperative tests designed to obtain a quantitative measure of the adhesion of bituminous films to mineral aggregates and resistance of these films to "stripping." While results of the cooperative tests have not been too encouraging, the subcommittee plans to continue its work on the problem, which is conceded to be difficult of solution.

In the field of aggregates, Subcommittee B-12 has completed its investigation of a suggested quality test for fine aggregate particles, based on abrasion under compression. For the information of interested Society members, a summary report on this investigation, "Crushing Tests of Sands," is appended to this annual report.

This report has been submitted to letter ballot of the committee, which consists of 144 members; 91 members have returned their ballots, of whom 82 have voted affirmatively and 1 negatively.

Respectfully submitted on behalf of the committee,

C. E. PROUDLEY,
Chairman.

B. A. ANDERTON,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-4 presented to the Society through the Administrative Committee on Standards the following recommendations:

Tentative Method of:
Test for Specific Gravity of Compressed Bituminous Mixtures (D 1188 - 51 T).

Tentative Specifications for:
Crushed Stone, Crushed Slag, and Gravel for Bituminous Concrete Base and Surface Courses of Pavements (D 692 - 51 T).

Revision of Tentative Specifications for:
Fine Aggregate for Sheet Asphalt and Bituminous Concrete Pavements (D 1073 - 49 T).

Tentative Revision of Standard Specifications for:
Granite Block for Pavements (D 59 - 39).

Withdrawal of Standard Specifications for:
Crushed Stone, Crushed Slag, and Gravel for Bituminous Concrete Base and Surface Courses of Pavements (D 692 - 49).

These recommendations were accepted by the Standards Committee on September 12, 1951, and the new and revised tentatives together with the tentative revision of D 59 - 39 appear in the 1951 Supplement to Book of ASTM Standards, Part 3.

APPENDIX I

CRUSHING TESTS OF SAND

SUMMARIZED BY D. L. BLOEM[1] ON BEHALF OF SUBCOMMITTEE B-12 OF COMMITTEE D-4

Recent work of Subcommittee B-12 on Structural Properties of Mineral Aggregates of ASTM Committee D-4 on Road and Paving Materials has been directed toward development of a test for structural strength of fine aggregates. Such a test would find chief application in connection with bituminous paving construction as an indication of the ability of the sand particles to withstand degradation under the impact and wear of traffic. A direct crushing test was considered by the subcommittee to offer promise of providing that information and it is with the investigation of such a test that this report deals.

Six laboratories of organizations represented on the subcommittee conducted independent tests to determine the feasibility of developing a significant crushing test. These were the laboratories of the Bureau of Public Roads, the South Atlantic Division of the U. S. Dept. of the Army, Corps of Engineers, the National Crushed Stone Assn. the National Sand and Gravel Assn., the National Slag Assn., and the North Carolina State Highway Commission.

Although the different laboratories worked entirely independently and were given free choice as to methods, the testing procedures used were very similar. In all cases, samples of sand of limited size range were subjected to pressure in a metal cylinder by means of a piston or pistons acting on one or both ends of the sample. The measure of breakdown was taken as the percentage of the original sample which would pass a sieve smaller than the minimum particle size in the original sample—generally the sieve upon which the sample was originally retained.

TEST METHODS

Variations in testing procedures are summarized in Table I. Diameters of the test cylinders ranged from 1.295 in. to 4.0 in. with three of the six laboratories using a 2.0 in. diameter. In three of the laboratories, the loading mechanism was so arranged that movable pistons applied pressure to both ends of the sample. In the remaining three laboratories the cylinder was closed at one end with a movable piston applying pressure only to the upper surface of the sample

Five of the laboratories used samples of fixed weight, ranging from 25 to 200 g. The sixth laboratory used a sample of constant bulk volume to reduce variations resulting from differences in specific gravity or void content of different sands.

Practices as to the amount of applied pressure varied among the laboratories. Two used different loads for different size fractions in an effort to produce equal or nearly equal losses for all sizes of the same material. Three laboratories used a fixed load of 2000 psi for all samples. One of these also made tests on one size using different loads to measure the effect of load on the loss produced. Another, in addition to using 2000 psi, made two preliminary tests at 1000 psi. The sixth laboratory made separate tests at 1000, 2000, and 3000 psi on each size.

Only two laboratories employed a specified uniform rate of loading, one of 16 psi per sec and the other a rate of compression of the sample of ¾ in. per min. The other laboratories, while not specifying a rate, attempted to provide the same loading conditions for all samples. Three of the laboratories released the load immediately upon reaching the specified pressure while the remaining three maintained the pressure for a fixed period of time before releasing it.

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[1] Assistant Director of Engineering, National Sand and Gravel Assn.

TEST RESULTS

Bureau of Public Roads:

A considerable number of crushing tests of fine aggregate were made at the Physical Research Branch of the Bureau of Public Roads, Washington, D. C., in an effort to measure the strength properties of sand particles. In one group of tests, summarized in Table II, samples of uniform materials of known hardness were compared. It will be noted that there was no consistent relationship between hardness and crushing resistance. The hardest material, quartz, with a Mohs hardness of 7, gave only slightly better performance than the very soft gypsum with a hardness of only 2. The materials of intermediate hardness, feldspar and limestone, showed better resistance than either the quartz or gypsum.

TABLE I.—SUMMARY OF TESTING TECHNIQUES EMPLOYED IN CRUSHING TESTS OF FINE AGGREGATE.

Laboratory	Diameter of Test Cylinder, in.	Plunger Action[a]	Size of Sample	Applied Load, psi	Rate of Loading	Time of Retention of Full Load, min
Bureau of Public Roads	1.295	Single	25 g.	Var.[b]	Unspecified	0
Corps of Engrs. U. S. Army, South Atlantic Div	2.0	Double	100 g.	Var.[c]	Unspecified	0
Nat'l Crushed Stone Assn	2.0	Single	100 g.	2000 psi[d]	Unspecified	1.0
Nat'l Sand and Gravel Assn	4.0	Double	2 in. depth	2000 psi[e]	16 psi per sec	1.0
Nat'l Slag Ass'n	4.0	Single	200 g.	2000 psi	Uniform	0
N. C. State Highway Comm	2.0	Double	150 g.	1000 psi 2000 psi 3000 psi	¾ in. per min	0.5

[a] "Double" indicates plungers at both ends movable. "Single" indicates only upper plunger movable.
[b] Load varied from 900 psi to 10,000 psi to give approximately same loss for all sizes.
[c] Load varied from 1000 psi to 8000 psi to give approximately same loss for all sizes.
[d] Tests also made on No. 16 to 30 size using different loads, ranging from 318 to 11,140 psi.
[e] Two tests also made at 1000 psi.

TABLE II.—RESULTS OF CRUSHING TESTS ON SELECTED MATERIALS OF VARYING HARDNESS

Tests made by the Physical Research Branch, Bureau of Public Roads, Washington, D. C.

See Table I for description of testing method.

Losses are averages of tests on five individual sizes: No. 4 to 8, No. 8 to 16, No. 16 to 30, No. 30 to 50 and No. 50 to 100, of each of the pure, or nearly pure, mineral or rock types. Samples prepared by crushing and screening. Losses are percentages passing the sieve upon which the sample was originally retained.

Material	Hardness (Mohs Scale)	Crushing Loss, per cent
Quartz	7	43.1
Feldspar	6	38.1
Limestone	4	36.1
Gypsum	2	45.1

Extensive studies were also made by the Bureau of Public Roads in which 33 different sands were subjected to various tests for quality, including the crushing test. Comparisons of results are given in Table III where the sands have been grouped into three broad categories as siliceous, granitic and sedimentary, on the basis of predominating rock types.

It can be seen in Table III that no general relationship was found between crushing resistance and results of freezing and thawing, sulfate soundness or mortar-strength tests. This fact alone, of course, is no proof that the crushing test is not indicative of particle strength since none of the other tests is considered as being an accurate measure of that quality. Nevertheless, the fact that the siliceous sands which contained the hardest particles gave highest losses in the test suggests that other characteristics than particle strength exert an excessive influence on results. It has been suggested that the soft materials tend to produce a fine powder in the early phase of the test, producing a cushioning effect that prevents actual splitting or fracture of the larger pieces and hence reduces the loss.

U. S. Dept. of the Army, Corps of Engineers:

Crushing tests were made at the Materials

Testing Laboratory of the South Atlantic Division, U. S. Dept. of the Army, Corps of Engineers, on four fine aggregates—two types of crushed stone, a natural river sand, and Ottawa sand. For the crushed sands and the natural river sand, the aggregates were separated into three sizes—No. 4 to 16, No. 16 to 50 and No. 50 to 100—and these sizes tested separately. Three separate tests were made of each size of each aggregate. Additional tests were made of the natural river sand separated into five individual sizes—No. 4 to 8, No. 8 to 16, No. 16 to 30, No. 30 to 50 and No. 50 to 100—and of the Ottawa sand in the available sizes—No. 16 to 30, No. 30 to 50, and No. 50 to 100. For these tests of the more closely sized materials, only two duplicate tests were made. In all cases the load was progressively increased as the size decreased.

TABLE III.—COMPARISONS OF CRUSHING TEST RESULTS WITH OTHER TESTS FOR SAND QUALITY

Tests made by the Physical Research Branch, Bureau of Public Roads, Washington, D. C.

Sand	Description	Loss in 60 Cycles Freezing and Thawing, per cent[a]	Loss in 5 Cycles Na_2SO_4 Test[a]	Compression Strength of Mortar, psi[b]	Crushing Loss, per cent[a]
No. 1...	Siliceous	10.6	4.3	4900	20.3
No. 2...	Granitic	5.9	2.8	5070	18.2
No. 3...	Siliceous	3.4	4.2	4820	19.4
No. 4...	Siliceous	2.1	2.0	4570	18.4
No. 5...	Siliceous	2.1	4.0	5340	6.3
No. 6...	Sedimentary	7.5	5.7	4720	18.6
No. 7...	Sedimentary	3.6	3.6	4810	15.2
No. 8...	Sedimentary	6.2	3.0	4800	15.8
No. 9...	Siliceous	4.4	5.2	4800	27.0
No. 10...	Sedimentary	5.3	4.6	4760	13.6
No. 11...	Granitic	9.0	2.6	4950	22.7
No. 12...	Granitic	7.9	2.3	4300	12.0
No. 13...	Sedimentary	14.4	4.1	4910	17.9
No. 14...	Siliceous	3.6	7.1	4730	29.0
No. 15...	Siliceous	7.4	3.8	4460	16.4
No. 16...	Siliceous	4.7	5.0	4920	30.0
No. 17...	Sedimentary	8.3	5.7	4920	14.4
No. 18...	Siliceous	6.0	2.8	5030	23.0
No. 19...	Sedimentary	5.4	3.8	4880	16.8
No. 20...	Sedimentary	3.5	3.2	4480	19.1
No. 21...	Sedimentary	22.5	12.6	4600	30.2
No. 22...	Siliceous	8.4	3.8	4870	21.7
No. 23...	Sedimentary	11.9	5.2	4840	16.4
No. 24...	Granitic	3.4	2.8	4810	9.2
No. 25...	Siliceous	6.9	3.6	5190	23.0
No. 26...	Sedimentary	9.6	7.3	4880	24.4
No. 27...	Siliceous	3.8	4.2	4890	17.1
No. 28...	Siliceous	2.4	2.0	4360	21.9
No. 29...	Granitic	3.8	3.5	4660	13.6
No. 30...	Sedimentary	6.3	5.7	4200	15.5
No. 31...	Granitic	11.2	2.6	4250	22.8
No. 32...	Granitic	4.4	4.8	5160	20.1
No. 33...	Granitic	10.7	4.3	5400	26.7

[a] Weighted averages based on gradings of sands as received.

[b] Standard Method of Test for Measuring Mortar-Making Properties of Fine Aggregates (C 87 – 47), 1949 Book of ASTM Standards, Part 3, p. 754, except that all sands were separated into sizes and recombined to produce same grading.

TABLE IV.—RESULTS OF CRUSHING TESTS OF VARIOUS FINE AGGREGATES.

Tests made at South Atlantic Division Materials Testing Laboratory, U. S. Army Corps of Engineers, Marietta, Ga.

See Table I for description of testing method.

Test Size (Sieve Nos.)	Grading Per cent Individual Size[a]	Applied Test Load psi	Percentage Loss[b]			
			Test 1	Test 2	Test 3	Avg.
CRUSHED GRANITE						
No. 4 to 16....	37	2000	27.9	27.2	26.6	27.2
No. 16 to 50...	43	4000	27.8	28.4	28.3	28.2
No. 50 to 100..	20	8000	42.3	42.5	44.0	42.9
Weighted Avg..			30.8	30.8	30.8	30.8
CRUSHED LIMESTONE						
No. 4 to 16...	37	2000	40.2	33.6	43.3	39.0
No. 16 to 50...	43	4000	34.7	34.9	37.0	35.5
No. 50 to 100..	20	8000	49.5	49.3	49.5	49.4
Weighted Avg..			39.7	37.3	41.8	39.6
NATURAL RIVER SAND						
No. 4 to 16...	37	2000	17.1	16.6	19.6	17.8
No. 16 to 50...	43	4000	15.2	14.6	15.3	15.0
No. 50 to 100..	20	8000	23.7	22.9	23.5	23.4
Weighted Avg..			17.6	17.0	18.5	17.7
No. 4 to 8....	20	1000	12.1	14.4	...	13.2
No. 8 to 16...	17	3000	29.6	32.4	...	31.0
No. 16 to 30...	21	3000	24.2	26.3	...	25.2
No. 30 to 50...	22	5000	22.2	23.2	...	22.7
No. 50 to 100..	20	8000	23.4	23.4	...	23.4
Weighted Avg..			22.1	23.7	...	22.9
OTTAWA SAND						
No. 16 to 30...	[c]	3000	2.0	2.2	...	2.1
No. 30 to 50...	[c]	5000	13.8	11.8	...	12.8
No. 50 to 100..	[c]	8000	13.6	14.7	...	14.2

[a] Based on average of fine aggregate grading in Standard Specification for Concrete Aggregates (C 33 – 49) 1949 Book of ASTM Standards, Part 3, p. 715. Percentages for largest test size include permissible quantity larger than No. 4 sieve. Percentages for smallest test size include all material smaller than that size.

[b] Percentage passing sieve upon which test sample was originally retained.

[c] Grading and weighted average for Ottawa sand not included since full range of sizes not available.

Individual test results are given in Table IV along with weighted average values based on the mean of the grading limits given in ASTM Standard Specification for Concrete Aggregates (C 33 - 49).[2] It can be seen that repeated tests on a given size of the same material in general checked very well and that weighted losses of a given material were in excellent agreement.

As would be expected, the testing of the five closely graded sizes of the natural river sand resulted in a higher weighted average loss than was obtained when the three broader size groups were used. The No. 4 to 8 size, for example, in the closely graded tests was sieved over the No. 8 sieve after crushing and would therefore show a higher loss than when sieved over the No. 16 sieve as was done when the test size was No. 4 to 16.

There is strong evidence in Table IV that factors other than particle strength had a pronounced influence on the losses produced. The crushed granite and crushed limestone which consisted of sharp, angular particles had much higher losses for all sizes than the natural river sand which was composed of well-rounded particles. By far the lowest losses for comparative sizes were obtained with the Ottawa sand in which the particles were very nearly spherical in shape. These data suggest that particle shape may have more influence on crushing loss than the actual strength of particles.

National Crushed Stone Association:

Comparative crushing tests of a natural river sand and two manufactured sands were made at the laboratory of the National Crushed Stone Association in Washington, D. C. Duplicate tests were made on each of five separated sizes—No. 4 to 8, No. 8 to 16, No. 16 to 30, No. 30 to 50, and No. 50 to 100—using a constant load of 2000 psi. The magnitude of load was established on the basis of preliminary tests of the No. 16 to 30 size of the natural river sand in which eleven different loads were tried. These ranged from 318 to 11,140 psi and produced losses from about 2 to 51 per cent.

Percentage losses for individual sizes and weighted average losses for the three sands, based on the average grading given in ASTM Specification C 33 - 49,[2] are shown in Table V.

Here again the reproducibility of the test was very good as evidenced by the excellent agreement between duplicate tests of a given size, and between weighted average losses for repeated tests on the same sand. However, there is again evidence that the particle shape of the sands exerted a large influence on test results. Losses for the natural river sand, composed of well-rounded grains, were very much lower than for either of the manufactured, angular sands. It is extremely doubtful that this large difference in loss can be attributed to differences in particle strength.

TABLE V.—RESULTS OF CRUSHING TESTS OF VARIOUS FINE AGGREGATES

Tests made at laboratory of the National Crushed Stone Association. Washington, D. C.
See Table I for description of testing method.

Test Size (Sieve Nos.)	Grading Per cent Individual Sizes[a]	Percentage Loss[b]		
		Test 1	Test 2	Average
POTOMAC RIVER SAND				
No. 4 to 8	20	31.5	33.5	32.5
No. 8 to 16	17	24.5	23.5	24.0
No. 16 to 30	21	21.0	20.0	20.5
No. 30 to 50	22	8.0	7.5	7.8
No. 50 to 100	20	8.0	8.0	8.0
Weighted Avg.		18.2	18.2	18.2
DOLOMITIC LIMESTONE SAND				
No. 4 to 8	20	52.0	48.5	50.2
No. 8 to 16	17	33.0	35.0	34.0
No. 16 to 30	21	27.0	24.5	25.8
No. 30 to 50	22	21.0	20.0	20.5
No. 50 to 100	20	15.0	16.0	15.5
Weighted Avg.		29.3	28.4	28.8
TRAP ROCK (DIABASE) SCREENINGS				
No. 4 to 8	20	47.0	43.0	45.0
No. 8 to 16	17	37.0	39.0	38.0
No. 16 to 30	21	34.0	35.0	34.5
No. 30 to 50	22	31.5	30.0	30.8
No. 50 to 100	20	28.0	28.0	28.0
Weighted Avg.		35.4	34.8	35.1

[a] Based on average of fine aggregate grading in Standard Specification for Concrete Aggregates (C 33 - 49). Percentages for largest test size include permissible quantity larger than No. 4 sieve. Percentages for smallest test size include all material smaller than that size.

[b] Percentage passing sieve upon which test sample was originally retained.

National Sand and Gravel Association:

Two fine aggregates, a nearly pure silica bank sand and Ottawa silica sand, were subjected to crushing tests at the Research Foundation of the National Sand and Gravel Association at the University of Maryland. The only major departure from the procedures used by the other laboratories was the use of a sample of constant bulk volume rather than constant weight. This procedure was adopted to eliminate, in so far as possible, the effects of differences in boundary conditions which might arise because of variations in volume of sand resulting from differences in specific gravity and void content. The quantity of sand used was that required to fill the 4-in. diam. test cylinder to a depth of 2 in. The resulting weights of samples, in grams, ranged from 517 (for the No. 50 to 100 size) to 614 (for the No. 4 to 8 size) for the concrete sand, and from 590 (for the No. 50 to 100 size) to 635 (for the No. 16 to 30 size) for the Ottawa sand.

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[2] 1949 Book of ASTM Standards, Part 3, p. 715.

TABLE VI.—RESULTS OF CRUSHING TESTS OF TWO FINE AGGREGATES.

Tests made at the National Sand and Gravel Association Research Foundation, University of Maryland, College Park, Maryland.

See Table I for Description of testing method.

Test Size (Sieve Nos.)	Grading Per Cent Individual Sizes[a]	Compression of 2 in. Sample, in.			Percentage Loss					
					Through Full-Size Sieve[b]			Through Half-Size Sieve[c]		
		Test 1	Test 2	Avg.	Test	Test 2	Avg.	Test 1	Test 2	Avg.
NATURAL QUARTZ CONCRETE SAND										
No. 4 to 8	20	0.278	...	0.278	34.7	...	34.7	16.4	...	16.4
No. 8 to 16	17	0.238	...	0.238	23.4	...	23.4	10.3	...	10.3
No. 16 to 30	21	0.206	...	0.206	17.1	...	17.1	5.4	...	5.4
No. 30 to 50	22	0.157	0.159	0.158	8.9	8.2	8.6	2.8	2.4	2.6
No. 50 to 100	20	0.184	0.165	0.174	5.9	6.2	6.0	2.2	2.2	2.2
Weighted Avg.							17.6			7.2
OTTAWA SAND										
No. 16 to 30	[d]	0.049	...	0.049	0.5	...	0.5	0.03	...	0.03
No. 30 to 50	[d]	0.062	...	0.062	1.7	...	1.7	0.08	...	0.08
No. 50 to 100	[d]	0.088	...	0.088	0.8	...	0.8	0.15	...	0.15

[a] Based on average of fine aggregate grading in Standard Specification for Concrete Aggregates (C 33 – 49). Percentages for largest test size include permissible quantity larger than No. 4 sieve. Percentages for smallest test size include all material smaller than that size.

[b] Percentage passing sieve upon which test sample was originally retained.

[c] Percentage passing sieve with opening one-half as large as that upon which sample was originally retained.

[d] Grading and weighted average fo Ottawa sand not included since full range of sizes not available.

A 2000-psi load was used in these tests after previous trials with a 1000-psi load had shown losses which were considered too low to offer promise of distinguishing bebetween sands of varying particle strength. In general, only a single test was made on each size fraction of each sand but repeated tests were made on the two smaller sizes of the concrete sand as a check on reproducibility.

The test results, summarized in Table VI, bear out the indications of previously described investigations that particle shape has a pronounced influence on losses, independent of particle strength. In spite of the fact that the Ottawa sand and the concrete sand were mineralogically nearly identical, the former, which was composed of nearly spherical grains, gave losses only a fraction as large as those of the concrete sand which was considerably more angular. An indication of the difference in angularity is given by the fact that individual sizes of the Ottawa sand contained, on the average, only 43 per cent voids as compared with 49 per cent for the same sizes of the concrete sand.

In addition to losses through the original retaining sieve, losses were also determined through the half-size sieve and deflections of the samples during test were measured with an Ames dial. These measurements correlated well with losses through the full-size sieve and are not considered as offering any additional indication of particle strength.

TABLE VII.—RESULTS OF CRUSHING TESTS OF VARIOUS FINE AGGREGATES.
Tests made at the Research Laboratory of the National Slag Association, Walford, Lawrence County, Pa.
See Table I for description of testing method.

Test Size (Sieve Nos.)	Grading per cent Ind. Sizes[a]	Soundness Loss,[b] per cent	Percentage Loss in Crushing Test							
			Thru Full-Size Sieve[c]				Thru Half-Size Sieve[d]			
			Test 1	Test 2	Test 3	Avg.	Test 1	Test 2	Test 3	Avg.
2000-PSI TEST LOAD										
Sandyville Natural Sand										
No. 4 to 8	20	14.7	42.0	43.3	42.1	42.5	19.4	19.9	20.2	19.8
No. 8 to 16	17	10.7	28.1	28.9	26.3	27.8	12.6	13.8	11.6	12.7
No. 16 to 30	21	4.9	19.5	20.8	20.1	20.1	8.2	9.0	8.7	8.6
No. 30 to 50	42	2.7	8.9	8.2	9.3	8.8	3.4	2.9	2.6	3.0
Weighted Avg.		6.9	21.0	21.4	21.0	21.1	9.2	9.4	8.9	9.2
Thompson Natural Sand										
No. 4 to 8	20	38.6	41.8	45.3	41.9	43.0	20.3	22.8	20.7	21.3
No. 8 to 16	17	29.9	38.7	37.0	37.6	37.8	18.5	18.4	17.6	18.2
No. 16 to 30	21	13.3	29.4	29.8	28.9	29.4	14.9	15.1	14.6	14.9
No. 30 to 50	42	9.6	15.6	14.8	15.6	15.3	7.6	6.8	7.5	7.3
Weighted Avg.		19.6	27.7	27.8	27.3	27.6	13.5	13.7	13.3	13.5
Bessemer Limestone Sand										
No. 4 to 8	20	9.8	56.8	56.9	54.8	56.2	25.8	25.3	24.8	25.3
No. 8 to 16	17	5.7	47.0	48.1	47.8	47.6	17.7	18.3	18.4	18.1
No. 16 to 30	21	4.9	35.3	34.6	35.9	35.3	13.6	13.3	14.0	13.6
No. 30 to 50	42	2.3	28.3	28.9	28.2	28.5	9.1	9.2	9.2	9.2
Weighted Avg.		4.9	38.6	39.0	38.5	38.7	14.8	14.8	14.9	14.8
Republic Slag Sand										
No. 4 to 8	20	2.0	62.7	62.9	64.9	63.5	35.9	36.4	37.5	36.6
No. 8 to 16	17	1.9	58.5	57.2	57.4	57.7	29.5	29.1	29.0	29.2
No. 16 to 30	21	0.0	47.9	47.6	48.3	47.9	23.0	22.9	23.2	23.0
No. 30 to 50	42	0.3	39.2	40.1	41.3	40.2	14.6	15.2	15.1	15.0
Weighted Avg.		0.8	49.0	49.1	50.2	49.4	23.2	23.4	23.6	23.4
Ottawa Sand										
No. 20 to 30	[e]	...	1.5	1.3	0.9	1.2	0.3	0.3	0.3	0.3
No. 30 to 50	[e]	...	4.2	3.8	4.1	4.0	0.1	0.1	0.2	0.1
3000-PSI TEST LOAD										
Thompson Natural Sand										
No. 4 to 8	20	38.6	50.1	48.7	50.2	49.7	26.8	27.2	26.8	26.9
No. 8 to 16	17	29.9	42.1	45.2	42.4	43.2	22.7	23.3	23.2	23.1
No. 16 to 30	21	13.3	36.1	36.1	35.3	35.8	20.2	19.6	19.3	19.7
No. 30 to 50	42	9.6	21.2	21.9	21.9	21.7	10.6	11.1	11.2	11.0
Weighted Avg.		19.6	33.7	34.2	33.8	33.9	17.9	18.2	18.0	18.0
Ottawa Sand										
Nos. 20 to 30	(5)	...	2.4	2.7	1.9	2.3	0.2	0.3	0.2	0.2

[a] See footnote[a], Table IV.
[b] Percentage loss in five cycles of the sodium sulfate test made in accordance with ASTM Method C 88 - 46 T.
[c] See footnote[b], Table VI.
[d] See footnote[c], Table VI.
[e] See footnote[c], Table IV.

TABLE VIII.—RESULTS OF CRUSHING TESTS OF VARIOUS FINE AGGREGATES.

Tests made at the laboratory of the North Carolina State Highway and Public Works Commission, Raleigh, N. C.
See Table I for description of testing method.

Test Size (Sieve Nos.)	Grading per cent Ind. Sizes[a]	Los Ang. Abrasion Loss, per cent	Percentage Loss in Crushing Test					
			1000 psi Load		2000 psi Load		3000 psi Load	
			Full Sieve[c]	Half Sieve[d]	Full Sieve	Half Sieve	Full Sieve	Half Sieve
QUARTZ SAND—NO. 1								
No. 8 to 16	37		17.4	5.3	37.8	16.3	48.3	24.0
No. 16 to 30	21		10.0	2.3	22.2	7.1	35.3	14.6
No. 30 to 50	42		5.3	...	14.5	...	22.8	...
Weighted Avg.		...	10.8		24.8		34.9	
QUARTZ SAND—NO. 2								
No. 8 to 16	37		23.5	1.0	53.7	11.0	56.3	20.3
No. 16 to 30	21		5.7	0.3	15.4	2.8	34.3	10.8
No. 30 to 50	42		1.0	...	5.6	...	14.8	...
Weighted Avg.		...	10.3		25.5		34.2	
QUARTZ SAND—NO. 3								
No. 8 to 16	37		14.5	3.9	41.2	16.0	52.3	25.1
No. 16 to 30	21		8.8	2.0	22.3	7.6	37.6	15.8
No. 30 to 50	42		6.1	...	14.9	...	24.8	...
Weighted Avg.		50	9.8		26.2		37.7	
METAMORPHIC SAND—NO. 4								
No. 8 to 16	37		21.0	7.0	34.8	16.2	41.6	20.7
No. 16 to 30	21		11.8	3.4	26.5	10.8	36.2	17.8
No. 30 to 50	42		12.5	...	25.2	...	32.3	...
Weighted Avg.		40	15.5		29.0		36.6	
GRANITE SCREENINGS—NO. 5								
No. 8 to 16	37		37.2	16.0	44.9	20.3	58.5	30.1
No. 16 to 30	21		31.0	13.3	41.3	20.4	46.6	24.8
No. 30 to 50	42		26.7	...	35.7	...	42.1	...
Weighted Avg.		24	31.5		40.3		49.1	
GRANITE SCREENINGS—NO. 6								
No. 8 to 16	37		41.0	12.2	55.9	21.1	58.2	23.6
No. 16 to 30	21		23.9	7.4	37.3	13.9	42.8	17.2
No. 30 to 50	42		17.8	...	25.8	...	36.2	. .
Weighted Avg.		28	27.7		39.3		45.7	
GRANITE SCREENINGS—NO. 7								
No. 8 to 16	37		28.9	8.7	46.3	19.2	55.5	25.0
No. 16 to 30	21		20.0	5.1	32.7	11.8	40.5	17.1
No. 30 to 50	42		10.3	...	21.9	...	31.3	...
Weighted Avg.		30	19.2		33.2		42.2	
GRANITE SCREENINGS—NO. 8								
No. 8 to 16	37		40.3	14.9	60.0	31.1	64.2	32.8
No. 16 to 30	21		32.0	14.1	42.9	21.2	51.4	27.9
No. 30 to 50	42		19.4	...	28.2	...	37.6	...
Weighted Avg.		31	29.8		43.0		50.3	

TABLE VIII—(*Continued*)

Test Size (Sieve Nos.)	Grading per cent Ind. Sizes[a]	Los Ang. Abrasion Loss,[b] per cent	Percentage Loss in Crushing Test					
			1000 psi Load		2000 psi Load		3000 psi Load	
			Full Sieve[c]	Half Sieve[d]	Full Sieve	Half Sieve	Full Sieve	Half Sieve
			GRANITE SCREENINGS—NO. 9					
No. 8 to 16	37		41.6	17.3	55.7	29.7	61.2	34.9
No. 16 to 30	21		32.6	11.8	44.4	20.4	51.3	26.6
No. 30 to 50	42		14.9	...	25.4	...	37.2	...
Weighted Avg		43	28.5		40.6		49.0	
			GRANITE SCREENINGS—NO. 10					
No. 8 to 16	37		45.8	18.4	61.2	32.4	67.8	38.0
No. 16 to 30	21		24.7	7.4	38.3	14.4	49.7	22.9
No. 30 to 50	42		13.6	...	24.3	...	30.7	...
Weighted Avg		59	27.9		40.9		48.4	
			LIMESTONE SCREENINGS—NO. 11					
No. 8 to 16	37		35.0	10.3	48.2	18.2	58.1	26.5
No. 16 to 30	21		28.6	9.6	38.5	14.9	50.0	21.7
No. 30 to 50	42		26.8	...	36.9	...	46.3	...
Weighted Avg		18	30.2		41.4		51.5	
			LIMESTONE SCREENINGS—NO. 12					
No. 8 to 16	37		13.6	2.2	24.8	6.8	37.5	13.1
No. 16 to 30	21		5.4	1.5	11.8	3.9	24.3	9.7
No. 30 to 50	42		5.7	...	16.3	...	23.7	...
Weighted avg		20	8.6		18.5		28.9	
			LIMESTONE SCREENINGS—NO. 13					
No. 8 to 16	37		48.1	15.8	61.3	28.8	66.2	32.6
No. 16 to 30	21		36.9	17.0	48.0	25.6	49.5	28.6
No. 30 to 50	42		32.0	...	43.1	...	48.9	...
Weighted Avg		27	39.0		50.9		55.4	
			LIMESTONE SCREENINGS—NO. 14					
No. 8 to 16	37		54.4	19.7	69.2	34.2	75.5	42.1
No. 16 to 30	21		46.4	13.0	51.9	25.9	60.6	34.4
No. 30 to 50	42		25.9	...	40.8	...	44.5	...
Weighted Avg		41	40.7		53.6		59.4	
			LIMESTONE SCREENINGS—NO. 15					
No. 8 to 16	37		43.3	15.8	58.5	26.9	68.0	35.5
No. 16 to 30	21		24.3	8.5	39.2	17.3	49.9	24.5
No. 30 to 50	42		14.4	...	27.7	...	39.9	...
Weighted Avg		46	27.2		41.5		52.2	
			LIMESTONE SCREENINGS—NO. 16					
No. 8 to 16	37		49.8	24.9	62.2	35.7	65.9	38.6
No. 16 to 30	21		47.2	28.8	53.6	35.5	54.9	38.0
No. 30 to 50	42		50.0	...	52.9	...	57.9	...
Weighted Avg		50	49.3		56.5		60.2	

[a] Based on average of fine aggregate grading in Standard Specifications for Concrete Aggregates (C 33 - 49), 1949 Book of ASTM Standards, Part 3, p. 715. Percentages for largest test size include permissible quantity larger than No. 4 sieve. Percentages for smallest test size include all material smaller than that size.

ASTM Standard Method of Test for Abrasion of Coarse Aggregate by Use of the Los Angeles Machine (C 131 - 47), 1949 Book of ASTM Standards, Part 3, p. 740. Tests made on parent coarse aggregates.

[c] Percentage passing seive upon which test sample was originally retained.

[d] Percentage passing sieve with opening one-half as large as that upon which sample was originally retained.

National Slag Association:

Crushing tests were made at the National Slag Assn's Research Laboratory in Walford, Lawrence County, Pa., on two natural sands, a crushed limestone, a crushed slag, and on available sizes of Ottawa sand. Three repeat tests were made using a 2000 psi load on each of four sizes—No. 4 to 8, No. 8 to 16, No. 16 to 30, and No. 30 to 50—for the four sands where gradings encompassed those sizes and on two sizes—No. 20 to 30 and No. 30 to 50—of the Ottawa sand. Similar tests were made using a 3000 psi load on one of the natural sands and on the No. 20 to 30 size of Ottawa sand.

Individual losses and weighted average results, based on the grading shown in A.S.T.M. Specifications C 33, are shown in Table VII for the samples sieved both on the original retaining sieve and on the half-size sieve. All samples were also sieved over the quarter-size sieve but relationships were so similar to those for the two larger sieves that the data have not been shown. Also shown in Table VII are results of 5-cycle sodium sulfate soundness tests made in accordance with ASTM Method: C 88 - 46T.[3]

It can be seen that there was no relationship between sulfate soundness loss and resistance to crushing. However, as stated before, this fact in itself is not significant in judging the crushing test since the soundness test is not intended to measure particle hardness.

Here again, the angular aggregates appear to be at a serious disadvantage in the crushing test as evidenced by the much higher losses for the two crushed sands than for the two natural sands. Comparisons among the various sands line up about the same for losses through either the full-size or half-size sieve except that the limestone sand compares more favorably with the natural sands on the latter basis. The following comparison of the relationships between the two methods of measuring loss, based on the tests made at a pressure of 2000 psi, may be of interest:

Fine Aggregate	Ratio, loss through half-size sieve to loss through full-size sieve
Sandyville natural sand	0.44
Thompson natural sand	0.49
Bessemer limestone sand	0.38
Republic slag sand	0.47

The fact that the ratio for the limestone sand was significantly lower than for the other three materials suggests a difference in nature of breakdown. Apparently the limestone particles tended to fracture into relatively large pieces and had less tendency to degrade into fine powder than did the other three materials. The data are too limited to indicate whether or not the half-size to full-size loss ratio, or that ratio considered along with the magnitude of the losses themselves, might be used as an indication of inherent hardness. It seems quite unlikely, however, that such relationships could be entirely isolated from the effects of particle shape.

As in the tests made by other laboratories, the losses for Ottawa sand were much lower than for corresponding sizes of the other materials. The use of the 3000-psi test load resulted in increased loss but appeared to offer no advantage over the 2000-psi load. Repeated tests on the same material showed excellent reproducibility.

North Carolina State Highway and Public Works Commission:

Studies of crushing tests at the laboratory of the North Carolina State Highway and Public Works Commission involved a total of 16 different fine aggregates including four natural sands, six crushed granites and six crushed limestones. Three sizes of each fine aggregate—No. 8 to 16, No. 16 to 30, and No. 30 to 50—were subjected to crushing under three loadings—1000, 2000, and 3000 psi. After test, each sample was sieved over all smaller sieves down to, and including, the No. 50 and the percentage passing each sieve was determined. Results are summarized in Table VIII where losses have been expressed as percentages passing the sieve upon which the sample was originally retained and the sieve having an opening one half that large.

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[3] Tentative Method of Test for Soundness of Aggregates by Use of Sodium Sulfate or Magnesium Sulfate (C 88 - 46), 1949 Book of ASTM Standards, Part 3, p. 776.

This investigation supports the findings of the other laboratories in indicating that this type of crushing test offers little promise of measuring the strength of fine aggregate particles. Here again the influence of particle shape is suggested by the lower losses produced in the rounded natural sands as compared with the angular crushed materials. The relationship is illustrated in the following comparison of average losses for the three types of aggregate studied:

Aggregate Type	Number Tested	Average Loss, per cent		
		1000 psi Load	2000 psi Load	3000 psi Load
Natural sand	4	11.6	26.4	35.8
Granite screenings	6	27.4	39.5	47.5
Limestone screenings	6	32.5	43.7	51.3

The suggestion might naturally follow that evaluation of test results could be weighted to take particle shape into consideration. Actually, however, the angularity of both natural and crushed aggregates, even of the same mineral or geologic type, can vary over such a wide range that a reliable procedure for taking this factor into consideration would seem almost impossible of attainment, at least until a quantitative measure of angularity has been developed.

Study of Table VIII reveals no relationship between loss in the Los Angeles abrasion test of the parent material and loss in the crushing test of fine aggregate, even for materials of the same general type. Attempts to develop relationships involving comparison of losses under different loadings or losses through the full-size and half-size sieves were also unsuccessful.

It is of interest to note that a slightly wider range in losses for different materials of a given type was obtained for the 1000 psi loading than for either the 2000- or 3000-psi loading, as indicated in the following tabulation:

Aggregate Type	Number Tested	Range in Crushing Loss, Percentage points		
		1000 psi Load	2000 psi Load	3000 psi Load
Natural sand	4	5.7	4.2	3.5
Granite screenings	6	12.3	9.8	8.1
Limestone screenings	6	40.7	38.0	31.3

The converse might be expected because of the greater magnitude of losses for the higher loads and hence the probability of greater differences. The fact that such was not the case suggests that whatever property is measured in this test, whether it be angularity, surface texture, strength of particle or a combination of these, is indicated more readily by the smaller loads.

CONCLUSION

While the data from individual investigations reported herein were too limited to permit drawing firm conclusions, taken collectively they seem to offer little hope that a crushing test of fine aggregate can be made to give an accurate measure of over-all particle strength. While the test is relatively simple and quick and gives excellently reproducible results in repeated tests of the same material, the losses produced appear to be affected too much by particle shape, and possibly other factors, to give a significant indication of structural strength.

APPENDIX II

PROPOSED METHOD OF TEST FOR DISTILLATION OF COAL TAR, COAL-TAR PITCH, AND SIMILAR TARS AND PITCHES[1]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

Scope

1. This method of test covers the distillation of coal tar, coal-tar pitch, and similar tars and pitches.

Outline of Method

2. A 100-g sample is distilled in a 300-ml flask at a controlled rate. The weights of distillate fractions at a series of specified temperatures and of residue at the maximum specified temperature are determined. The residue (and distillates) may be reserved for additional tests.

Apparatus

3. The apparatus shall consist of the following:

(*a*) *Flask.*—A side-neck distillation flask, as shown in Fig. 1, conforming to the following dimensions:

Diameter of bulb, outside	86.0 ± 1.5 mm
Diameter of neck, inside	22.0 ± 1.0 mm
Diameter of tubulature, inside	10.0 ± 0.5 mm
Height of flask, outside	131.0 ± 1.5 mm
Vertical distance, bottom of bulb, outside, to horizontal tangent at tubulature, inside	93.0 ± 1.5 mm
Length of tubulature	220 ± 5 mm
Angle of tubulature	73 ± 2 deg
Thickness of tubulature wall.	1.0 to 1.5 mm

(*b*) *Condenser Tube.*—A tapered glass condenser, as shown in Fig. 3, conforming to the following dimensions:

Outside diameter of small end	12.5 ± 1.5 mm
Outside diameter of large end.	28.5 ± 3.0 mm
Length	360 ± 4 mm
Length of tapered part	100 ± 5 mm

(*c*) *Shield.*—A shield of galvanized iron, lined with $\frac{1}{8}$-in. asbestos. It shall be provided with a transite board cover, of the form and dimensions shown in Fig. 2, used to protect the flask from air currents and to prevent radiation. If desired, a shield not provided with mica windows may be used.

(*d*) *Gauze.*—Two sheets of 20-mesh wire gauze made of No. 26 B & S gage Nichrome wire. They shall be 125 to 150 mm in diameter or square.

(*e*) *Shield for Burner.*—A metal shield 95 to 105 mm in inside diameter, approximately 100 mm in height, to be suspended from the ring support for the flask, as shown in Fig. 3.

(*f*) *Receivers.*—The receivers shall be tared and have a nominal capacity of 50 to 125 ml.

(*g*) *Balance and Weights.*—The balance and weights shall be accurate to 0.05 g.

(*h*) *Thermometer.*—The thermometer shall be an ASTM High Distillation Thermometer, total immersion, having a range of −2 to +400 C, and conforming to the requirements for Thermometer 8C as prescribed in the Standard Specifications for ASTM Thermometers (ASTM Designation: E 1).[2]

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[1] This proposed method is under the jurisdiction of the ASTM Committee D-4 on Road and Paving Materials.

This method is a proposed revision of the Standard Method of Test for Distillation of Tar Products Suitable for Road Treatment (D 20 - 30) which is under consideration by Committee D-4.

[2] 1950 Supplement to Book of ASTM Standards, Part 3.

NOTE.—When conducting a distillation to temperature above approximately 370 C, the temperature of the bulb approaches a critical range in the glass. Accordingly, the ice point of a thermometer, which has been used under such conditions, shall be checked before the thermometer is used again.

Preparation of Sample

4. Agitate the sample, immediately before testing, to insure a complete mixture. If warming is necessary, take care to avoid the loss of volatile material.

Dehydration of Sample

5. If water is present in excess of 2.0 per cent, dehydrate a representative portion of the sample before distillation, in accordance with the Standard Method of Test for Water in Creosote (ASTM Designation: D 370).[2]

Apparatus Assembly

6. (*a*) Suspend the burner-shield from a tripod or ring and place two sheets of the specified wire gauze on the tripod or ring. With the flask shield in place, support the flask with its bulb resting on the wire gauze, as shown in Fig. 3. Connect the tubulature of the flask to the condenser by a tight cork joint, with the tubulature projecting 30 to 50 mm through the cork. Support the condenser by a suitable clamp. The distance from the neck of the flask to the outlet end of the condenser should be not more than 540 nor less than 500 mm.

(*b*) Insert the thermometer through a cork in the neck of the flask, with the top of the bulb level with the lowest point of the juncture between the tubulature and neck of the flask. The axis of the bulb of the flask through the center of the neck should be vertical and the thermometer aligned on this axis.

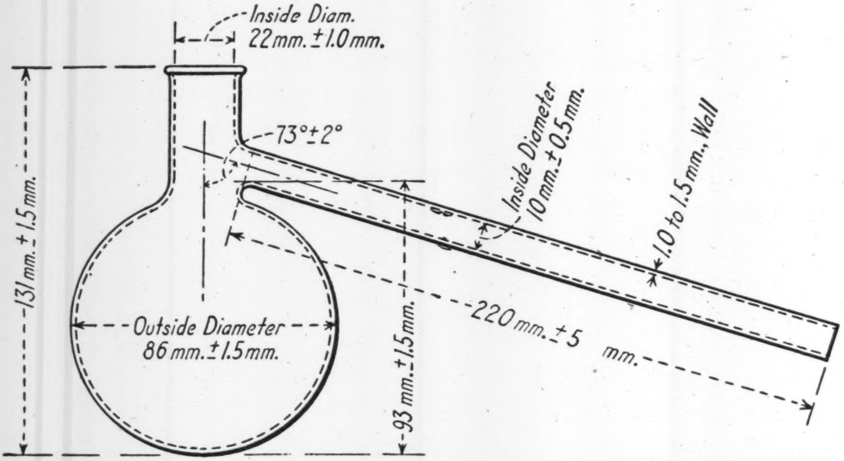

Inside Diam. 22mm. ± 1.0mm.
73° ± 2°
Inside Diameter 10mm. ± 0.5mm.
1.0 to 1.5mm., Wall
131mm. ± 1.5mm.
Outside Diameter 86mm. ± 1.5mm.
93 mm. ± 1.5mm.
220mm. ± 5 mm.

FIG. 1.—Distillation Flask.

Procedure

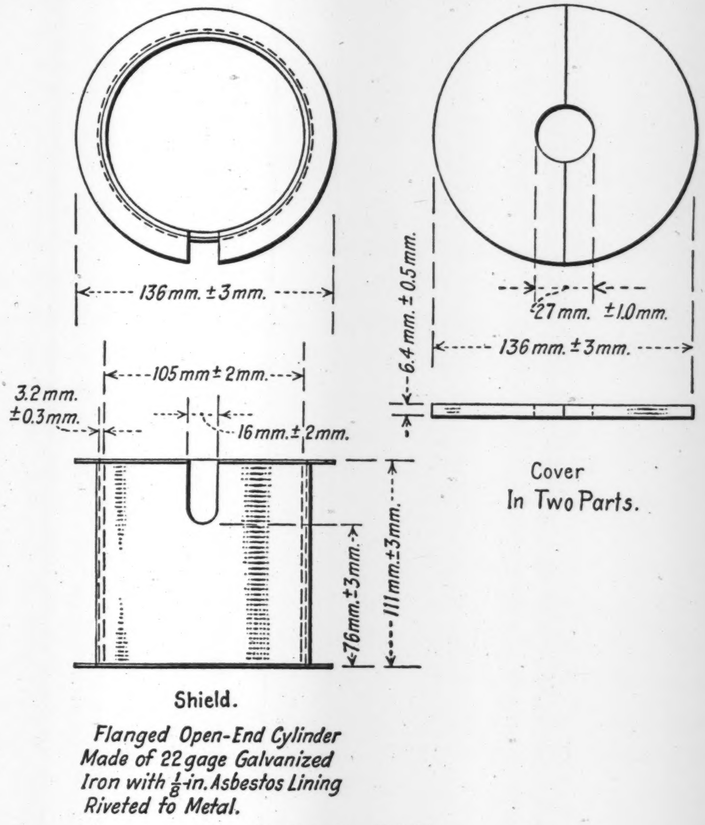
7. (*a*) Weigh the flask to the nearest 0.05 g, and then weigh 100.0 + 0.1 g of the sample into the flask. Assemble the apparatus and apply heat so that the first drop falls from the end of the condenser within 5 to 15 min. Adjust the rate of distillation within 2 min after the first drop, and subsequently maintain that rate so that from 50 to

70 drops per min fall from the end of the condenser. Collect the fractions of distillate, in receivers previously weighed to the nearest 0.05 g, at the temperatures designated by the specifications. Change the receivers as the thermometer indicates the maximum temperature for each fraction. When the maximum specified temperature of the test is indicated by the thermometer, immediately remove the flame and shield cover. Drain any oil remaining in the condenser tube, after at least 5 min, into the receiver containing the last fraction. Weigh the receivers containing the fractions to the nearest 0.05 g.

(*b*) Allow the residue to remain in the flask, with the cork and thermometer in place, for at least 5 min, or until no vapors are visible. Remove the cork and thermometer. Weigh the flask containing the residue to the nearest 0.05 g.

(*c*) During the progress of the distillation, allow the thermometer to remain in its original position. Make no correction for the emergent stem of the thermometer.

(*d*) If tests of the residue are required, replace the cork and the thermometer in the flask, and lower the thermometer until its bulb is in the liquid residue. Then incline the flask so that the residue will flow around the sides, and collect any condensed vapors that may be on the sides of the flask. Mix the



Shield.

Flanged Open-End Cylinder Made of 22 gage Galvanized Iron with $\frac{1}{8}$-in. Asbestos Lining Riveted to Metal.

Cover
In Two Parts.

FIG. 2.—Shield.

contents of the flask until they are homogeneous. When the residue has cooled to a temperature of 100 to 150 C, pour it into the desired testing equipment or into a suitable receptacle. Cover receptacle. If the residue becomes so cool that it cannot be poured readily from the flask, reheat it carefully until it is completely liquid and at a temperature not exceeding 150 C, preferably by holding the bulb of the flask over a wire gauze heated by a burner or by immersion of the bulb in a suitable bath at a temperature not exceeding 150 C.

TABLE I.—TEMPERATURES AT WHICH TAR FRACTIONS SHALL BE CUT TO CORRECT DISTILLATION TEMPERATURES FOR DIFFERENT ALTITUDES.

(Corrections made to the Nearest Degree Centigrade.)

Elevation Above Sea Level, Ft.	Fractionation Temperatures for Various Altitudes, deg. Cent.					
0	170	200	235	270	300	355
1000	169	198	233	268	298	353
1500	168	198	232	267	297	352
2000	167	197	231	266	296	351
2500	167	196	230	265	295	350
3000	166	195	230	264	294	349
3500	165	195	229	263	293	348
4000	165	194	228	263	292	347
4500	164	193	227	262	291	346
5000	164	192	226	261	290	345
5500	163	192	225	260	289	344
6000	162	191	225	260	288	343

(*e*) If any of the fractions contain water, determine the volume of water in each fraction and calculate the weight of oil distillate in each fraction, assuming that 1 ml of water weighs 1 g A convenient method for determining the amount of water is to transfer the fraction, after weighing, completely to a suitable tube or cylinder graduated in 0.1 ml, and adding an amount of benzene sufficient to cause a clear separation between the oil solution and water.

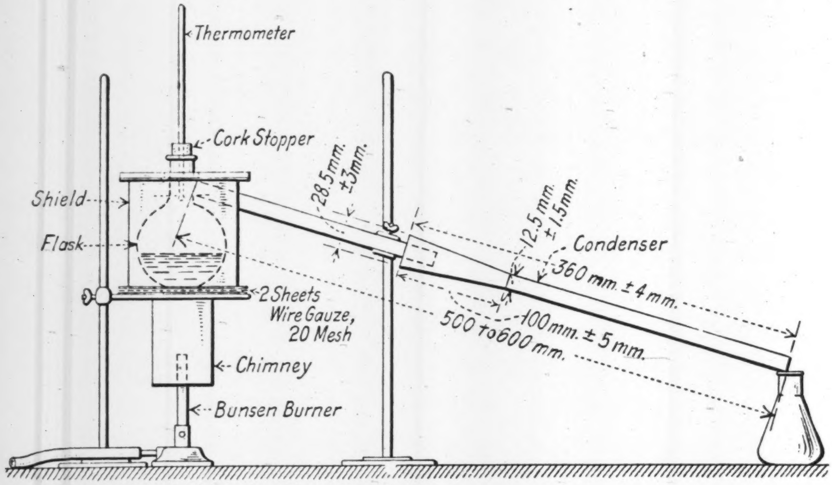


FIG. 3.—Distillation Apparatus Assembly.

NOTE.—This cannot be done when further tests on the distillate fraction are required.

Report

8. (*a*) Report the results of the distillation test in percentages by weight of water-free material, expressed to the nearest 0.1 per cent. The following fractions are usually recorded:

Up to 170 C
170 to 235 C
235 to 270 C
270 to 300 C
Residue at 300 C

At times other fractions are required, such as 300 to 355 C, and residue at 355 C. It is customary to report percentages of total distillate to a series of temperatures corresponding to the maximum temperatures of the fractions.

(*b*) If the elevation at which the distillation is to be made exceeds 1000 ft, correct the temperatures at which the fractions are taken, in accordance with Table I.

REPORT OF COMMITTEE D-5

ON

COAL AND COKE*

Committee D-5 on Coal and Coke held meetings in Atlantic City, N. J., on June 27, 1950, and in Cincinnati, Ohio, on March 5, 1951. The Advisory Subcommittee held one meeting on the latter date during the ASTM Spring Meeting Week.

During the year, four members were added to the committee and one member resigned, resulting in a total membership of 61, of whom 18 are classified as consumers, 18 as producers, and 25 as general interest members.

Chairman W. A. Selvig has continued his active participation as delegate to the Working Party on Classification, Coal Committee, Economic Commission for Europe; he attended one meeting in Geneva, Switzerland, during the year.

ADOPTION OF TENTATIVE AS STANDARD

Committee D-5 recommends that the Tentative Method of Test for Grindability of Coal by the Hardgrove-Machine Method (D 409 - 37 T)[1] be approved for reference to letter ballot of the Society for adoption as standard with the following revision:

Appendix.—Delete the entire Appendix which gives conversion values for Hardgrove to ball-mill grindability indexes.

Footnote Reference.—In the scope clause add a reference to the following new footnote: "Another method of test for the grindability of coal, Tentative Method of Test for Grindability of Coal by the Ball-Mill Method (D 408 - 37 T), was published as tentative by the Society from 1937 to 1950."

WITHDRAWAL OF TENTATIVE

The committee recommends that the Tentative Method of Test for Grindability of Coal by the Ball-Mill Method (D 408 - 37 T)[1] be withdrawn. This action is recommended in view of the almost universal acceptance of the Hardgrove-machine method (D 409 - 37 T)[1] for commercial use. It is recognized that the ball-mill method has merit and that considerable data have been accumulated by use of this method. For this reason, provision has been suggested for continuing a reference to the Society's long publication of the ball-mill method as tentative, such reference to consist of a footnote in the Hardgrove-machine method which has been recommended for adoption as standard.

EDITORIAL CHANGES

Standard Anthracite Sizing Specifications, adopted by the Anthracite Committee of the Production Control Plan for the Anthracite Industry on July 28, 1947, have been superseded by somewhat different specifications as set forth in the Anthracite Standards Law of the Commonwealth of Pennsylvania, effective September 1, 1949. The differences particularly affect terminology. Accordingly the following editorial changes, which do not affect the test procedure, are recommended in the Standard Method of Test for Size of Anthracite (D 310 - 34)[1]:

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* Presented at the Fifty-fourth Annual Meeting of the Society, June 18-22, 1951.

[1] 1949 Book of ASTM Standards, Part 5.

Section 2.—Change the third sentence, by the addition of the italicized words and the deletion of those in brackets, to read as follows: "Screens mounted in hardwood box frames 16 to 20 in. square are satisfactory for testing [chestnut] *nut,* pea, buckwheat, *and rice* sizes of anthracite."

Section 3 (a).—Change this section to read as follows: "The screen openings specified in the Anthracite Standards Law of the Commonwealth of Pennsylvania, effective September 1, 1949, as standard anthracite sizing specifications, are as follows:"

In the table change the following size designations: "chestnut" to read "nut"; No. 1 to read "Buckwheat"; "No. 2 Buckwheat (Rice) to read "Rice." Also in the table delete the last size designation' 'No. 3 Buckwheat (Barley)" and its respective screen opening specifications.

Section 6.—In the table showing approximate sizes of laboratory samples make corresponding changes in size designations to those indicated above for the table in Section 3 (*a*).

TENTATIVE CONTINUED WITHOUT REVISION

The committee recommends that the Tentative Method of Sampling and Analysis of Coal for Volatile Matter Determination in Connection with Smoke Ordinances (D 980 - 48 T)[1] be continued as tentative without revision during the coming year, since it is felt that insufficient experience has been accumulated to warrant other action.

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee VII on Pulverizing Characteristics of Coal (J. C. Witt, chairman) held no meetings during the year but did undertake a letter ballot of its membership regarding the disposition of the two long-standing tentative methods for grindability of coal (D 408 - 37 T and D 409 - 37 T). As a result the subcommittee recommended, and Committee D-5 now concurs, that the Tentative Method of Test for Grindability of Coal by the Ball-Mill Method (D 408 - 37 T) be withdrawn and that the Tentative Method of Test for Grindability of Coal by the Hardgrove-Machine Method (D 409 - 37 T) be adopted as standard with certain minor revisions, as set forth earlier in this report.

Subcommittee XIII on Coal Sampling (H. F. Hebley, chairman) held no meetings during the year, but a special section of this subcommittee devoted to automatic sampling, under the chairmanship of R. P. Liversidge, met twice. At the last session, on March 5, 1951, this subcommittee considered jointly with members of the Prime Mover's Committee, Power Station Chemistry Subcommittee, of the Edison Electric Institute a detailed test scheme which the EEI group proposes to apply to one coal, using one make of automatic sampler. The scheme is designed to eliminate the question of machine bias in sampling and to provide reliable information on variability (based on analysis of variance) not only in the automatically collected increments, but in hand increments of several sizes collected simultaneously by partitioning the stream on a stopped belt. The ultimate aim is to promote standards which will permit least cumbersome automatic sampling for large users of crushed or fine coal.

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[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM headquarters.

Subcommittee XV on Plasticity and Swelling of Coal (C. C. Russell, chairman) held a meeting on March 6 and considered in detail the results of a number of Gieseler plasticity tests run by various laboratories. Tests were run on identical samples of each of two different silicones having viscosities within the range common to this test. Further work toward elimination of unwanted variables was suggested at the meeting.

A proposed revision of the Standard Test for Free-Swelling Index of Coal (D 720 - 46) was submitted and will be circulated for detailed consideration. Briefly, this revision would permit measurement of the actual coke button size to obtain an index number where the button shape does not conform to any standard profile of the present test.

Subcommittee XXI on Methods of Analysis (O. W. Rees, chairman) met on March 5 and considered further its revision of methods for the ultimate analysis of coal and coke. Suggestions for further revision were studied and will be incorporated in a redraft for circulation to the committee.

A first draft of a proposed procedure for the determination of mineral CO_2 was also submitted and will be circulated.

This report has been submitted to letter ballot of the committee, which consists of 61 voting members; 57 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

W. A. SELVIG,
Chairman.

C. H. SAWYER,
Secretary

REPORT OF COMMITTEE D-6

ON

PAPER AND PAPER PRODUCTS*

Committee D-6 on Paper and Paper Products held two meetings during the year: on June 29, 1950, at Atlantic City, N. J., and on February 23, 1951 at New York, N. Y. The Advisory Subcommittee held three meetings during the year: on June 28, 1950, at Atlantic City, N. J., on December 8, 1950, and February 23, 1951, in New York, N. Y.

A new committee was established during the year to be known as the Joint D-6—D-9 Committee. This committee replaces the old joint committee on pH problems and is set up to coordinate all of the activities of common interest to these two ASTM committees. The D-6 members on this committee are Hubbell Lathrop and P. F. Wehmer.

B. W. Scribner has replaced Roger Griffin, who is no longer a member of the committee, as one of the D-6 representatives on the Joint ASTM-TAPPI Committee on Paper Testing Methods.

The by-laws were revised during the year to modernize them and to regulate more closely the activities of the committee.

At the present time, Committee D-6 consists of 87 members, of whom 69 are voting members; 28 are classified as producers, 26 as consumers, 25 as general interest, and 8 as consulting members.

NEW TENTATIVES

The committee recommends that the following four methods be accepted for publication as tentative, as appended hereto:[1]

Tentative Methods of Test for:

Chloride Content of Paper and Paper Products,
Water-Soluble Matter in Paper,
Lint of Paper Towels, and
Ring Crush of Paperboard.

ADOPTION OF TENTATIVES AS STANDARD

The committee recommends that the following two tentative methods be approved without revision for reference to letter ballot of the Society for adoption as standard:

Tentative Methods of Test for:

Creasing Paper for Permeability Tests (D 1027 - 49 T), and
Ply Separation of Combined Container Board (D 1028 - 49 T).

REVISION OF TENTATIVES

The committee recommends for approval and continuation as tentative the following revised tentative methods, as appended hereto:[2]

Tentative Method of Test for:

Analytical Filter Papers (D 981 - 48 T), and
Water Vapor Permeability of Paper and Paperboard (D 988 - 48 T).

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends that the following three tentative methods which have stood for two years without revision be continued as tentative:

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* Presented at the Fifty-fourth Annual Meeting of the Society, June 18–22, 1951.

[1] The new tentatives were accepted by the Society and appear in the 1951 Supplement to Book of ASTM Standards, Part 4.

[2] The revised tentatives were accepted by the Society and appear in the 1951 Supplement to Book of ASTM Standards, Part 4.

Tentative Methods of Test for:

Puncture and Stiffness of Paperboard, Corrugated and Solid Fiberboard (D 781 - 44 T).—A revised method for puncture is still under investigation.

Stretch of Paper and Paper Products under Tension (D 987 - 48 T).—Possible modification of the method for stretch to adapt it to creped paper is under study.

Fiber Analysis of Paper and Paperboard (D 1030 - 49 T).—Consideration is being given to the removal of certain tests from the method for fiber analysis and their inclusion in an appendix as special tests.

The remaining tentative methods under the jurisdiction of Committee D-6, although requiring no revision, are also recommended for continuation as tentative.

The recommendations appearing in the report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Paper Testing Methods (M. S. Kantrowitz, chairman) held three meetings during the year. The subcommittee has been active in developing new methods of test and has also continued its critical study of various methods of test for paper and paper products that have been adopted by other agencies such as the Technical Association of the Pulp and Paper Industry. The subcommittee prepared three of the four new methods of test which are being recommended for publication as tentative, namely, Chloride Content of Paper and Paper Products, Water-Soluble Matter in Paper, and Lint of Paper Towels. One of the two tentative methods being recommended for adoption as standard (namely, D 1027) and tentative Methods D 981 and D 988 for which revision is recommended are also under the jurisdiction of this subcommittee.

Test methods to determine the following properties are being given consideration by the subcommittee: erasability, moisture expansivity, gloss, pinholes, color fastness to light, and zinc and cadmium pigments.

In addition, studies are being made which may lead to proposed revisions in the Standard Methods of Test for Opacity of Paper and Paper Products (D 589 - 44), Ply Adhesion of Paper (D 825 - 48), Conditioning Paperboard, Fiberboard, and Paperboard Containers for Testing (D 641 - 49), Conditioning Paper and Paper Products for Testing (D 685 - 44), Bursting Strength of Paper (D 774 - 46), Wet Tensile Breaking Strength of Paper and Paper Products (D 829 - 48), Resistance of Paper to Passage of Air (D 726 - 48), Turpentine Test for Grease Resistance of Paper (D 722 - 45), Time of Penetration by Water of Sized Paper and Paper Products (D 779 - 46), and Moisture in Paper, Paperboard, and Paperboard and Fiberboard Containers (D 644 - 44).

Subcommittee II on Significance of Test Methods (L. S. Reid, chairman) held three meetings during the year. It is expected that the revision of the Monograph on Paper and Paperboard; Characteristics, Nomenclature and Significance of Tests will be completed by fall.[4]

Subcommitee III on Specifications for Paper (P. F. Wehmer, chairman) held one meeting during the year. The subcommittee is working on a specification for kraft paper for multi-wall bags.

Subcommittee IV on Container Board (W. B. Lincoln, Jr., chairman) held two meetings during the year and is currently working on the following test procedures: Flat crush test, Immersion number, and Wet bursting strength.

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[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

[4] Published as STP No. 60-A.

The subcommittee prepared one of the four new methods of test that are being recommended for publication as tentative, namely, Ring Crush of Paperboard. Tentative Method D 1028 - 49 T, for which adoption as standard is being recommended, is also under the jurisdiction of this subcommittee. Studies are also being made which may result in proposed revisions in Standard Method of Sampling Paper and Paper Products (D 585 - 42).

This report has been submitted to letter ballot of the committee, which consists of 69 voting members; 47 members returned their ballots, of whom 39 voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

W. R. WILLETS,
Chairman.

R. H. CARTER,
Secretary.

REPORT OF COMMITTEE D-7

ON

WOOD*

A meeting of Committee D-7 on Wood was held in Chicago, Ill., on March 13, 1951. On March 12 meetings of the Subcommittees on Specifications for Timber and on Wood Preservatives, and a joint meeting of the Subcommittee on Fire-Retardant Wood with the corresponding committee of the Am. Railway Engineering Assn. were held. Progress on the work of a number of the other subcommittees was carried on through correspondence during the year.

Committee D-7 has taken cognizance of the organization of ISO Technical Committee TC55-Timber (Sizing, Defects), and has recommended that Committee D-7 be represented, at least on a liaison basis. The chairman of Committee D-7 has been authorized to represent Committee D-7 on ISO Technical Committee TC55 with power to act for the committee.

NEW TENTATIVE

Committee D-7 recommends the publication as tentative of the Proposed Method of Test for Methoxyl Groups in Wood, as appended hereto.[1]

ADOPTION OF TENTATIVE AS STANDARD

The committee recommends that the Tentative Specifications for Creosoted End-Grain Wood Block Flooring for Interior Use (D 1031 - 49 T)[2] be approved for reference to letter ballot of the Society for adoption as standard without revision.

TENTATIVE REVISION OF STANDARD

The committee recommends the publication of a tentative revision of the Standard Definition of Terms Relating to Wood (D 9 - 30)[2] in the form of new Tentative Nomenclature of Domestic Hardwoods and Softwoods, as appended hereto,[3] which is intended to replace the present Standard Names for Structural Timbers of D 9.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends that the remaining tentatives under its jurisdiction be retained as tentative, pending further study and contemplated revision.

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Specifications for Timber (Lyman W. Wood, chairman).—Active work is under way on the revision of the Standard Specifications for Round Timber Piles (D 25 - 37). A revised draft was reviewed in detail at the March 12 meeting. The revision will be completed during the coming year.

Because further study now under way

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* Presented at the Fifty-fourth Annual Meeting of the Society, June 18-22, 1951.

[1] The new tentative was accepted by the Society and appears in the 1951 Supplement to Book of ASTM Standards, Part 4.

[2] 1949 Book of ASTM Standards, Part 4.

[3] The tentative revision, in the form of new tentative, was accepted by the Society and appears in the 1951 Supplement to Book of ASTM Standards, Part 4.

[4] The letter ballot vote of the committee was favorable; the results of the vote are on record at ASTM Headquarters.

on the basic principles of structural grading may result in some changes in the Tentative Methods for Establishing Structural Grades of Lumber (D 245 - 49 T), it is recommended that these methods be retained as tentative until such revisions are completed.

Subcommittee II on Laminated Timber (F. J. Hanrahan, chairman).—Pending results of further research at the Forest Products Laboratory on accelerated tests for adhesives, it was voted to continue as tentative the Tentative Method of Test for Integrity of Glue Joints in Laminated Wood Products for Exterior Service (D 1101 - 50 T).

Subcommittee IX on Methods of Test (L. J. Markwardt, chairman).—Methods of test for fiberboards are now also under consideration by the Subcommittee on Mechanical Wood Technology of the Food and Agriculture Organization of the United Nations and unified procedures are expected to be developed by this group. Pending further comment and study, it is recommended that the Tentative Methods of Test for Evaluating the Properties of Building Boards (D 1037 - 49 T) be continued another year as tentative.

Research has continued on the improvement of a tension-parallel-to-grain test specimen as a modification of this test in Standard Methods of Testing Small Clear Specimens of Wood (D 143 - 50). The modified specimen that has given promising results is loaded by shear through notched supports at the ends instead of wedge grips. By eliminating slippage at the grips, the time of test is materially reduced, while satisfactory failure in the reduced section is still obtained.

Subcommittee X on Nomenclature and Definitions (R. R. Cahal, chairman).—A number of changes in botanical and common tree names have been made over a period of years by the U. S. Department of Agriculture. Subcommittee X has made special study of these changes to correlate them with the "Standard Names for Structural Timbers" as presented in ASTM Standard D 9 - 30. In addition, nomenclature for hardwoods has been prepared and the list of softwoods extended. As a result of this study, the Proposed Nomenclature of Domestic Hardwoods and Softwoods, as appended hereto,[2] is recommended for publication as a tentative revision of Definitons D 9 - 30.

Subcommittee XII on Fire-Retardant Wood (W. H. Fulweiler, chairman).—A joint meeting was held on March 12 with the corresponding committee of the AREA. Coordination with AREA is being continued to secure data on experience and classification of exposures with respect to requirements for fire-retardant treatments. It is hoped that satisfactory classification can be established in the near future.

Subcommittee XIV on Methods of Chemical Analysis (G. J. Ritter, chairman).—In keeping with the objective of establishing methods for the complete chemical analysis of wood and following the adoption of nine methods as tentative in 1950, a Method for Test of Methoxyl Groups in Wood was approved by the subcommittee and recommended for adoption as tentative. Other methods are also in preparation. It is planned to coordinate this program with that of the new ASTM technical committee of the Society on cellulose and cellulose derivatives now being organized.

This report has been submitted to letter ballot of the committee, which consists of 70 voting members; 52 members returned their ballots, of whom 50 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

L. J. Markwardt,
Chairman.

REPORT OF COMMITTEE D-8

ON

BITUMINOUS WATERPROOFING AND ROOFING MATERIALS*

During the year, Committee D-8 on Bituminous Waterproofing and Roofing Materials held two meetings: one in Atlantic City, N. J., on June 28, 1950, the other in Cincinnati, Ohio, on March 7, 1951.

The committee suffered the loss of one member, H. P. Pearson, through resignation.

The membership consists of 73 voting members, composed of 46 producers, 11 consumers and 16 general interest members.

New Tentative

Committee D-8 recommends that the proposed Methods of Testing Asphalt-Base Emulsions for Use as Protective Coatings for Built-up Roofs, as appended hereto,[1] be accepted for publication as tentative.

Adoption of Tentative as Standard

Specifications for Sieve Analysis of Granular Mineral Surfacing for Asphalt Roofing and Shingles (**D 1001 – 48 T**).[2]—The committee recommends that these specifications be approved for reference to letter ballot of the Society for adoption as standard.

Adoption of Tentative Revisions as Standard

The committee recommends that the tentative revisions pertaining to Section 2 and Table I, as submitted in June, 1950, of each of the following specifications be approved for reference to letter ballot of the Society for adoption as standard:

Standard Specifications for:

- Asphalt Shingles Surfaced with Mineral Granules (D 225 – 46),[3]
- Wide Selvage Asphalt Roofing Surfaced with Mineral Granules (D 371 – 46),[3] and
- Asphalt Siding Surfaced with Mineral Granules (D 699 – 46).[3]

Editorial Change

The committee recommends the following editorial changes in the *Standard Methods of Sampling and Testing Felted and Woven Fabrics Saturated with Bituminous Substances for Use in Waterproofing and Roofing* (**D 146 – 47**).[2] The present wording of Section 7 in Specifications for Woven Cotton Fabrics Saturated with Bituminous Substances for Use in Waterproofing (**D 173 – 44**) and Sections 16 and 20 of Methods D 146 leads to confusion as to whether the weight of fabric in bituminous saturated fabric is the bone-dry weight of the extracted fabric or whether the weight of the oven-dried extracted fabric is corrected for moisture regain as described in Section 2 of the General Methods of Testing Woven Textile Fabrics (**D 39 – 39**). An editorial change in Section 20 of Methods D 146 will eliminate this confusion; make the following revisions:

Section 20.—Change the boldface heading from "Weight" to "Thread Count" and change the section to read as follows: "20. Woven Fabrics shall be tested for number of threads per inch in accordance with Section 7 of the Standard General Methods of Testing Woven Textile Fabrics (ASTM Designation: D 39)."

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* Presented at the Fifth-fourth Annual Meeting of the Society, June 18–22, 1951.

[1] The new tentative was accepted by the Society and appears in the 1951 Supplement to Book of ASTM Standards, Part 3.

[2] 1949 Book of ASTM Standards, Part 3.

[3] 1950 Supplement to 1949 Book of ASTM Standards, Part 3.

Section 20 (b).—Renumber as Section 21. Add a boldface heading "Felt Number" and delete the present italicized "Felted Fabrics."

Section 21.—Renumber as Section 22.

TENTATIVES CONTINUED WITHOUT REVISION

Committee D-8 recommends that the following tentatives, which have been published by the Society for two or more years, be continued without revision since they are being reviewed for possible revisions to be recommended before the next Annual Meeting:

Tentative Methods of Testing Asphalt Rool Roofing, Cap Sheets and Shingles (**D 228 - 48 T**),

Tentative Recommended Practice for Accelerated Weathering Test of Bituminous Materials (**D 529 - 39 T**), and

Tentative Methods of Sampling Bituminous Materials (**D 140 - 49 T**) which is now under discussion with Committee D-4 on Road and Paving Materials for extensive revision since the two committees share joint jurisdiction over this tentative.

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

This report has been submitted to letter ballot of the committee, which consists of 73 members; 45 members have returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee.

A. J. STEINER,
Chairman.

G. W. ROBBINS,
Secretary.

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[4] The letter ballot vote of the committee was favorable; the results of the vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE D-9
ON
ELECTRICAL INSULATING MATERIALS*

Committee D-9 on Electrical Insulating Materials held two meetings during the year and will hold a third during the Annual Meeting of the Society in June. These meetings were held in Cincinnati, Ohio, on November 15 to 17, 1950, and in Washington, D. C., on March 28 to 30, 1951.

At these meetings action was taken on several new methods, new recommended practices, revisions of existing tentatives, and changes in some of the standards under the jurisdiction of Committee D-9. There are some projects still under way which will be mentioned later in the report.

Recommendations Affecting Standards

As a result of the year's work, the committee is submitting one new tentative, revision of one standard, tentative revisions in two standards, revision of four tentatives, and is recommending adoption as standard of four tentatives and the withdrawal of two tentatives. The standards and tentatives affected, together with the recommended revisions, are presented in detail in the Appendix to this report.

These recommendations have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[1]

Activities of Subcommittees

Subcommittee I on Insulating Varnishes, Paints, and Lacquers (A. H. Haroldson, chairman) is working on specifications for various types of varnishes. The Methods of Testing Varnishes (D 115 – 48 T) will be extended to include pH measurements, compatability, and salt water or immersion tests. The methods also will be classified either as control or performance tests. The control tests are intended to be used as a guide for uniformity and identification and the performance tests to determine the performance of the varnish for application work.

The Section on Heat Reactive Laminating Varnishes has completed a study of various methods for the determination of set time of laminating varnishes. Round-robin tests will be conducted on the various methods now being used in the industry. It should be possible to adopt the most applicable method extant using manual detection of the end point.

The Section on Silicone Insulating Varnishes has drafted proposed methods of testing silicone varnishes which include procedures for drainage time, drying time, and oil resistance, and dielectric strength. Round-robin tests will be conducted on these, as well as on the effect of sea water and dielectric strength.

The round-robin testing program for chemical resistance, which had been planned for the determination of deformation, creepage, and moisture resistance has been delayed because a study will first be made of the results of similar tests which are being conducted in Canada.

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* Presented at the Fifty-fourth Annual Meeting of the Society, June 18–22, 1951.

[1] The letter ballot vote of the committee was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee IV on Insulating Oils (E. A. Snyder, chairman).—A report on the activities of this subcommittee is appended to this report.

Subcommittee V on Ceramic Products (R. W. Orr, chairman) is preparing a revision of the Standard Methods of Testing Steatite Used as Electrical Insulation (D 667 – 44). This revision will include conditioning procedures for resistivity, power factor, and dielectric constant, based upon the conclusions drawn from the round-robin tests on these properties under humid conditions. Methods of test for thermal expansion and for resistivity, power factor, and dielectric constant at elevated temperatures are also being studied.

There is considerable interest in test methods for porous ceramic materials of the type used for insulators in electron tubes. The Joint Electron Tube Engineering Council has designated a representative to serve on Subcommittee V for this work.

Subcommittee VI on Solid Filling and Treating Compounds, (K. G. Coutlee, chairman) prepared the proposed Tentative Methods of Testing Hydrocarbon Waxes Used as Electrical Insulation, appended hereto.[1a]

In order to increase the effectiveness of the subcommittee activities, the sections of the subcommittee have been reorganized as follows:

Section	Title	Chairman
A.....	Electrical Properties	E. W. Greenfield
B.....	Chemical Properties	H. H. Coffman
C.....	Physical Properties	H. Schindler
D.....	Specifications	A. H. Postle
E.....	Scope and Significance	R. H. Titley

The newly organized sections will review the Methods of Testing Solid Filling and Treating Compounds Used for Electrical Insulation (D 176 – 44) to bring these methods up to date.

A special task group was appointed to review the use of casting resins and make recommendations for subcommittee action. As a result of the task group report, the question of consideration of casting resins is being submitted to letter ballot of Subcommittee VI.

Subcommittee VII on Varnished Fabrics (R. W. Chadbourn, chairman) has reviewed in detail the Tentative Methods of Testing Varnished Cloths and Varnished Cloth Tapes Used in Electrical Insulation (D 295 – 49 T) and Tentative Specifications for Black Bias-Cut Varnished Cloth Tape Used for Electrical Insulation (D 373 – 50 T) and has made extensive revisions, many of a minor nature. In Methods D 295, the sampling procedure was altered to provide for a minimum of two sample rolls, the dial micrometer method of measuring thickness was added, and the dielectric strength test procedure altered to provide for the use of $\frac{1}{4}$-in. electrodes for testing both cloths and tapes. A revised method for determining insulation resistance was adopted. The scope of Specifications D 373 was extended to include cloths, as well as tapes, and the classification of these changed from "greasy" and "tacky" to "greasy," "dry," and "tacky."

The subcommittee is still studying the problem of possible revision of the specification requirements for dielectric strength of varnished cloth tapes under 6 per cent elongation. More test data will be required before a decision can be reached. The round-robin tests to check the adequacy of the existing temperature-elongation formula in Specifications D 373 have been completed and the results are under study.

[1a] The new tentative was accepted by the Society and appears in the 1951 Supplement to Book of ASTM Standards, Part 6.

Proposed revisions in the Standard Methods of Testing Flexible Varnished Tubing Used for Electrical Insulation (D 350 – 48) and Tentative Specifications for Flexible Treated Cotton and Rayon Sleeving Used for Electrical Insulation (D 372 – 47 T), based on new methods adopted by the National Electrical Manufacturers Assn., are being studied by the subcommittee.

Significance statements for power factor and dielectric constant of varnished cloths and tapes and for conditioning for varnished glass fabrics and tapes have been developed and approved.[1b]

The subcommittee has completed its round-robin investigation of heat-aging of varnished glass fabrics and has developed a test procedure covering both oleoresinous-treated and silicone-treated fabrics. Consideration is being given to the matter of low-temperature requirements for glass tapes, particularly for use as cable insulation.

The subcommittee is pursuing its investigation of the degree of correlation between the conductance and tensile methods of determining corrosion resulting from the use of pressure-sensitive adhesive tapes on copper wire at high humidities (see Tentative Methods of Testing Pressure-Sensitive Adhesive Tapes Used for Electrical Insulation (D 1000 – 48 T)). Active work has begun on the development of test methods for plastic pressure-sensitive tapes. This will involve the investigation of many properties peculiar to this type of material.

Subcommittee VIII on Insulating Papers (E. G. Ham, chairman) assisted in the organization of the new general Joint Committee of D-6 and D-9, which has been authorized by both committees.

Progress has occurred in the refinement of a number of the procedures, which are recognized as being somewhat imperfect, in the Methods of Sampling and Testing Untreated Paper Used in Electrical Insulation (D 202 – 50 T). In addition, an investigation has been made of a total chloride residue test procedure for paper which might be more sensitive to the measurement of very small amounts of chlorides than is the present procedure, which was only recently adopted by Committee D-6.

Several significance statements have been formulated which are to be added to Methods D 202 but none have been submitted to Committee D-9 for acceptance this year.

Subcommittee IX on Mica (E. O. Hausmann, chairman).—Four Sections of Subcommittee IX report as follows.

Section A on Natural Mica reports that no suitable photo transparencies have been prepared to date. After several years of attempting to make suitable samples, this project has been abandoned. Since the photo transparencies project has been discontinued, the preparation of standard samples has been discussed.

Comments were sent to the American Standards Association for transmittal to ISO concerning the proposals submitted to ISO/TC 56 on Mica. The committee disagreed with the selecting of mica standards according to source and color. It was felt that the difference between the visual classifications should be based on the extent of physical imperfections only.

The proposed revision of the Standard Methods of Measuring Mica Stampings Used in Electronic Devices and Incandescent Lamps (D 652 – 43), appended hereto[2], was received from Committee B-4.

Section B on Bonded Mica prepared the tentative revision of the Standard Methods of Testing Pasted Mica Used in Electrical Insulation (D 352 – 49).

Section C on Specifications is interested in the electrical method for testing mica for imperfections which has been developed by K. G. Coutlee. Section C will explore the utility of this test as a possible substitute for visual quality tests.

[1b] See Editorial Note, p. 377.

[2] The revised methods were accepted by the Society and appear in the 1951 Supplement to Book of ASTM Standards, Part 6.

Subcommittee XI on Significance of Tests (J. H. Palmer, chairman) submitted to the committee proposed changes in the recommended practice for writing statements as to the usefulness of tests of electrical insulating materials; also recommendations regarding the positioning of significance statements in the D-9 compilation. These recommendations are being submitted to letter ballot of Committee D-9.

Subcommittee XIII on Mechanical Tests has been terminated. In its place a direct liaison between the Advisory Committee and Committee E-1 will be maintained.

Subcommittee XIV on Conditioning (A. C. Webber, chairman).—A joint D-9 and D-20 group revised, and submitted to the Administrative Committee on Standards, the Methods of Conditioning Plastics and Electrical Insulating Materials for Testing (D 618 – 49 T). The revised methods were accepted on March 2, 1951. This revision includes a number of additional standard test temperatures and an added high humidity procedure for electrical tests. The Recommended Practice for Maintaining Constant Relative Humidity by Means of Aqueous Solutions (D 1041 – 49 T) has been editorially revised and submitted to Committees D-9 and D-20 for adoption as standard. Work has continued on a new specification for Servicing Units and Enclosures for Tests of Plastics and Insulating Materials at Other than Normal Temperatures to replace Specifications D 760 and D 761.

Section B is obtaining technical information on all methods of measurement of relative humidity, which will be organized and presented to the subcommittee as information.

This report has been submitted to letter ballot of the committee; 91 members returned their ballots, of whom 78 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

A. H. SCOTT,
Chairman.

E. A. SNYDER,
Vice-Chairman.

M. GUARNIER,
Recording Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting Committee D-9 presented to the Society through the Administrative Committee on Standards the recommendation that the Tentative Methods of Testing Varnished Cloths and Varnished Cloth Tapes Used in Electrical Insulation (D 295 – 51 T) be revised. This recommendation was accepted by the Standards Committee on November 30, 1951, and the revised methods appear in the 1951 Supplement to Book of ASTM Standards, Part 6.

APPENDIX

RECOMMENDATIONS AFFECTING STANDARDS FOR ELECTRICAL INSULATING MATERIALS

In this Appendix are given recommendations affecting certain standards and tentatives concerning electrical insulating materials which are referred to earlier in this report. The standards and tentatives appear in their present form in the 1949 Book of ASTM Standards, Part 6, and its 1950 Supplement, as indicated by the final number in the ASTM designation.

NEW TENTATIVE

Tentative Methods of Testing Hydrocarbon Waxes Used as Electrical Insulation:

Committee D-9 recommends that these methods of testing, prepared by Subcommittee VI, be accepted for publication as tentative, as appended hereto.[1]

REVISIONS OF TENTATIVES

Tentative Methods of Testing Varnish Used for Electrical Insulation (D 115 - 48 T):

Subcommittee I recommends the following revisions in these methods:

Section 1.—Revise the first sentence to read: "These methods cover tests for varnishes primarily intended to provide electrical, mechanical and chemical protection for electrical equipment."

New Section 2.—Add a new Section 2 on "Classification and Definition" as follows, renumbering subsequent sections accordingly:

2. (*a*) *Alcohol Soluble Varnishes.*—Clear or colored varnishes which form films primarily by evaporation of solvents, and intended for application to electrical apparatus which may be exposed to mineral oils or related materials.

(*b*) *Oxidizing Air-Drying Varnishes.*—Clear or colored varnishes which form films at room temperatures, primarily by oxidation of the drying oil content, and intended for application to electrical apparatus which may be exposed to mineral oils and related materials.

(*c*) *Thermosetting Varnishes.*—Clear or colored varnishes which, when baked in depth, form infusible gels and intended for application to electrical apparatus which may be exposed to mineral oils or related materials.

(*d*) *Oxidizing, Baking Varnishes.*—Clear or colored varnishes which, when baked, form films primarily by a process of oxidation and intended for application to electrical apparatus which may be exposed to mineral oils and related materials.

(*e*) *Air-Drying Asphaltic Varnishes.*—Non-oil-proof, black asphaltic varnishes of the benzine soluble class which contain little or no drying oils and which form films primarily by evaporation of the solvent.

(*f*) *Silicone Varnishes.*—Clear baking varnishes with good thermal stability at temperatures within the range of 180 to about 250 C. These varnishes are usually used in conjunction with mica, glass, fibers, asbestos and similar inorganic materials.

(*g*) *Potting and Embedding Varnishes.*—Varnishes which are applied in liquid state and which may be polymerized in place without appreciable loss of solvent or components.

(*h*) *Thermosetting Laminating Varnishes.*—Clear or colored varnishes containing synthetic resins which, when heated, are converted by a chemical reaction into a rigid composition. They are used for impregnating and coating materials which, when built up into a laminate, may be pressed between metal plates by the application of heat and pressure to form a solid structure.

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[1] The new tentative was accepted by the Society and appears in the 1951 Supplement to Book of ASTM Standards, Part 6.

Section 2 (b).—Revise this paragraph to require the use of copper strips 1½ in. in width.

Tentative Methods of Testing Varnished Cloths and Varnished Cloth Tapes Used in Electrical Insulation (D 295 – 50 T):

Subcommittee VII recommends revision of these methods, altering the sampling procedure to provide for a minimum of two sample rolls, adding the dial micrometer method of measuring thickness, and altering the dielectric strength test procedure to include the use of ¼-in. electrodes for testing cloths and tapes. The revised methods are appended hereto.[2]

Tentative Specifications for Black Bias-Cut Varnished Cloth Tape Used for Electrical Insulation (D 373 – 50 T):

Revisions in these specifications, prepared by Subcommittee VII, are recommended as appended hereto.[3]

Tentative Methods of Testing Nonrigid Polyvinyl Tubing (D 876 – 50 T):

Subcommittee III recommends the following revision of Methods D 876:

Section 1.—Revise the first sentence to read: "These methods cover procedures for the testing of general purpose (Grade A) and high temperature (Grade C)[4] non-rigid polyvinyl chloride tubing, or its copolymers with other materials for use as electrical insulation."

Section 15 (a).—Change to read as follows:

15. (*a*) ***Oven***.—The oven shall conform to the following requirements:

(*1*) The design shall be such that heated air passes through the specimen chamber and is exhausted without being recirculated.

(*2*) Provision shall be made for suspending specimens, preferably vertically, without bending and without touching each other or the sides of the chamber. The specimen chamber shall be so designed or the oven so compartmented that air passing over any specimen shall not come in contact with other specimens in the oven.

(*3*) The temperature at any point along the length of the specimens shall not vary more than ±1 C from the specified temperature.

(*4*) The heating medium shall be air at atmospheric pressure and the source of heat shall be external to the specimen chamber or chambers.

(*5*) The air flow shall be lengthwise along the specimens and shall be at the rate of 100 ± 10 in. per min.

Section 17 (a).—Change the first sentence to read: "Three specimens shall be suspended in the oven described in Section 15 (*a*)." Revise the third sentence to read as follows:

The specimens shall be maintained at the temperatures listed below for a period of 72 hr:

Grade A	100 ± 1 C
Grade B.................	130 ± 2 C

Section 19 (b).—Revise to read: "The oven shall conform to the requirements prescribed in Section 15 (*a*)."

Section 21.—In the first sentence revise the first two words "The specimens" to read "Three specimens." In the second sentence change the present reference to the oven described in Section 19 (*b*) to the oven described in Section 15 (*a*). Revise the fourth sentence to read as follows:

The specimens shall be maintained at the temperatures listed below for a period of 72 hr:

Grade A................	100 ± 1 C
Grade B................	130 ± 2 C

Section 23.—Delete the phrase "with vinyl acetate" in the last sentence of this section.

Section 26 (a).—Revise the first sentence to read as follows:

The test specimens shall be totally immersed in ASTM No. 3 high-swelling oil (Note) as described in the Tentative Methods of Test for Changes in Properties of Rubber and Rubber-

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[2] The revised methods were accepted by the Society and appear in the 1951 Supplement to Book of ASTM Standards, Part 6.

[3] The revised specifications were accepted by the Society and appear in the 1951 Supplement to Book of ASTM Standards, Part 6.

[4] Methods for testing Grade B will be included at a later date.

Like Materials in Liquids (ASTM designation: D 471) at temperatures listed below for a period of 4 hr:

Grade A	70 ± 1 C
Grade B	105 ± 1 C

Section 31.—In the second sentence revise "100 ± 2 C" to read "100 ± 1 C."

TENTATIVE REVISION OF STANDARD

Standard Methods of Testing Sheet and Plate Materials Used in Electrical Insulation (D 229 - 49):

The committee recommends the publication of the following as a tentative revision of this standard:

Section 27.—Add a new Paragraph (*c*), as an alternate method for testing dielectric strength parallel with the laminations, to read as follows:

(*c*) As an alternate method for tests parallel to the flat sides (parallel with laminations in laminated sheets) the test specimen shall be 2 by 3 in. in thickness. American Standard tapered pins (such as Morse, Brown & Sharpe, or Pratt & Whitney) having a taper of $\frac{1}{4}$ in. per ft., shall be used as electrodes.

For specimen thicknesses up to and including $\frac{1}{2}$ in., No. 3 American Standard tapered pins[5] 3 in. long and having a diameter at the large end of $\frac{7}{32}$ in., shall be used. Drill two $\frac{3}{16}$-in. diameter holes, centrally located, 1 in. apart, center to center, and perpendicular to the faces of the specimen. Ream holes to a sufficient depth to allow the pins to extend approximately $1\frac{1}{2}$ in. from the small end of the hole.

For specimen thicknesses over $\frac{1}{2}$ in. up to and including 2 in., No. 4 American Standard pins[5] 4 in. long and having a diameter at the large end of $\frac{1}{4}$ in., shall be used. Drill two $\frac{3}{16}$-in. diameter holes, centrally located, 1 in. apart, center to center, and perpendicular to the faces of the specimen. Ream holes to a sufficient depth to allow the pins to extend approximately 1 in. from the small end of the hole.

The electrodes shall be inserted after the conditioning of the specimen. Spheres of $\frac{1}{2}$-in. diameter placed on the extremities of the tapered pins will decrease the tendency to flash-over.

New Section 31.—Revise the present section heading to read "Dielectric Dissipation Factor and Dielectric Constant." Add a new paragraph to read as follows:

31. *Test Specimens.*—The test specimens shall be rectangular pieces sawed from the sheets and of sizes shown in the following table. The electrodes shall cover both sides of the sheet and consist of silver paint sprayed or painted on or of metal foil held in place by a thin layer of petrolatum. When the same specimen is used for Condition A and, for immersion in water and when metal foil electrodes are used, the metal foil electrodes shall be removed and petrolatum cleaned off with a suitable solvent before immersion. It is not necessary to remove silver paint electrodes before immersion.

Thickness, in.	Size of Specimen, in.
Up to and including $\frac{3}{64}$	2 by 2
Over $\frac{3}{64}$ to $\frac{3}{32}$ incl.	3 by 3
Over $\frac{3}{32}$ to $\frac{1}{4}$ incl.	4 by 4
Over $\frac{1}{4}$ to 2 incl.	4 by 8

Section 31.—Renumber as Section 32 and renumber subsequent sections accordingly. In Paragraph (*a*) revise "power factor" to read "dissipation factor."

Standard Methods of Testing Pasted Mica Used in Electrical Insulation (D 352 - 49):

The committee recommends the publication of the following as a tentative revision of this standard: To these methods add the following new sections on definitions and significance for the various tests with section numbers as indicated, renumbering subsequent sections accordingly:

Decrease in Thickness Under Pressure:

2. *Definition.*—The decrease in thickness under pressure test determines the change in thickness of pasted mica under specified conditions of high pressure and temperature.

Significance.—This test determines the deformation under load under conditions of heat and pressure that may be encountered during manufacture and use of electrical equipment

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[5] For information on tapered pins, see Kent's Mechanical Engineers Handbook, 12th edition, Design & Production Volume, pp. 14–15.

The test is suitable for acceptance tests and for manufacturing control when pasted mica sheet with maximum stability under heat and pressure is required.

Stability Under Heat and Pressure:

8. *Definition.*—The test for stability under heat and pressure determines whether the binder will exude and the mica splittings will slip or be displaced under the specified conditions of test.

9. *Significance.*—Proper size and placement of the splittings, correct pressing technique, and the use of a minimum quantity of a suitable binder in the manufacture of pasted mica will result in a sheet with the maximum stability. If the viscosity of the binder at the test temperature is too low or too much has been used, the binder will flow when pressure is applied. The hydrostatic pressure upon the edges of a splitting and the fluid friction on its surfaces due to the outward flow of the binder may cause a lateral displacement of the splittings. When the sheet has been properly manufactured, no appreciable exudation of binder or displacement of splittings should occur during the test. The test serves to indicate in a general way the degree of stability under other conditions of heat and pressure which may be present during the manufacture and use of electrical equipment. The test is suitable for acceptance tests and for manufacturing control when pasted mica sheet with the maximum stability under heat and pressure is required.

Mica or Binder Content:

15. *Definition.*—The mica or binder content of pasted mica relates the proportional weights of mica and organic binder contained in the material. Results are expressed in percentage loss in weight of the specimen as binder and the percentage of residue as mica.

16. *Significance.*—In the application for pasted mica for hot molding, commutator and flexible insulation, heating plates, and similar purposes, the mica or binder content is important to obtain certain electrical and physical characteristics. Physical properties such as the ability to hot mold, flexibility, rigidity, heat resistance, tackiness, and good adherence of the plates to each other are directly related to the binder content. Electrical properties, such as dielectric strength and resistivity, are a function of the mica content.

Molding Test:

22. *Definition.*—The molding test for pasted mica is defined as a measure of the ability of the sheet to hold its shape when molded.

23. *Significance.*—Under prescribed heating conditions, the binder used in molding plate mica softens sufficiently to allow the plate to become flexible and to be formed by bending and, with slight movement of the component mica splittings, to take a definite shape. After cooling, the degree of form retention, flaking, and buckling indicates the suitability of the pasted mica plate for the purpose intended. This test is of considerable value for quality control and acceptance tests.

Dielectric Strength:

29. *Definition.*—Dielectric strength of pasted mica is the voltage gradient at which the material fails as a dielectric by electrical breakdown. Results are expressed in volts per mil of thickness.

30. *Significance.*—In the applications for pasted mica for hot molding, commutator insulation, heating plates, and similar purposes pasted mica is subjected to electrical stresses in service which are a small fraction of the breakdown stresses determined by the short-time or step-by-step tests. The short-time and step-by-step tests are, however, well adapted for specification acceptance and factory control purposes in that they will detect spots, impurities, voids, and other defects which may render the material unsuitable in service.

Resistivity:

33. *Definition.*—Resistivity of pasted mica is that property of the material which enables it to resist the passage of direct current. It comprises Volume and Surface Resistivities and Insulation Resistance, definitions of which are given in ASTM Method D 257. The results are expressed in ohm-cm units and ohms, respectively.

34. *Significance.*—The resistivity of pasted mica is a measure of its effectiveness as an electrical insulator under the application of constant unidirectional voltage. It serves primarily to indicate the presence of impurities and moisture, and give information as to uniformity in processing. At this time this test is not recommended as a basis for acceptance or rejection.

ADOPTION OF TENTATIVES AS STANDARD

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Methods of Test for:

Rockwell Hardness of Plastics and Electrical Insulating Materials (D 785 - 48 T), jointly with Committee D-20,

Detection of Free Sulfur in Electrical Insulating Oils (D 989 - 48 T), and

Power Factor and Dielectric Constant of Natural Mica (D 1082 - 49 T).

Tentative Recommended Practices for:

Maintaining Constant Relative Humidity by Means of Aqueous Solutions (D 1041 - 49 T), jointly with Committee D-20.

REVISION OF STANDARD, IMMEDIATE ADOPTION

Standard Methods of Measuring Mica Stamping Used in Electronic Devices and Incandescent Lamps (D 652 - 43):

The committee recommends for immediate adoption revisions of these methods, as appended hereto,[2] and accordingly requests the necessary nine-tenths affirmative vote in order that these recommendations may be referred to letter ballot of the Society.

WITHDRAWAL OF TENTATIVES

Tentative Specifications for Phenolic Laminated Sheet for Radio Applications (D 467 - 44 T), and

Tentative Specifications for Round Phenolic Laminated Tubing for Radio Applications (D 616 - 41 T):

The committee recommends the withdrawal of these two tentatives since they will be superseded by the revised Tentative Specifications for Laminated Thermosetting Materials (D 709 - 49 T).[8]

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[8] Revised Specifications D 709 - 52 T were accepted on February 15, 1952, by the Administrative Committee on Standards.

REPORT OF SUBCOMMITTEE IV ON LIQUID INSULATION

The uninhibited mineral transformer oil studies, referred to in previous reports, have been in progress about eight years. During the past year, four transformers were opened and examined and the condition of the transformer evaluated in terms of the laboratory test data.

Among the tests used for the laboratory study of the oils have been:

(*a*) Sludge Accumulation Test,
(*b*) High Pressure (bomb) Test,
(*c*) Color,
(*d*) Neutralization Value, and
(*e*) The Interfacial Tension Test.

One transformer examined indicated a condition which was predicted only by the high-pressure oxidation test. Other transformers examined led to the conclusion that, although the laboratory tests were able to evaluate differences in the basic oxidation resistance of oils for transformer use, these differences did not necessarily show up in the practical use of the oil.

One published report by F. M. Clark entitled "Evaluation of Mineral Transformer Oils During Service—Part I" on this cooperative study was presented before the Society in 1949.[1] It is now planned to hold a symposium on this general subject in the fall of 1951, at which time three papers will be presented for discussion and publication. One of these papers (by F. M. Clark) will examine the relative merits of the various laboratory tests studied. The other two papers (R. G. Call and T. A. McConnell) will describe the condition of the transformers examined in an effort further to correlate the laboratory data obtained on the various oils tested with the actual condition of the transformers examined.

Four methods of laboratory test for the evaluation of inhibited oils were adopted for study from the large number submitted for consideration of Section B. These tests are:

1. A Sludge Accumulation Test,
2. A High-Pressure (bomb) Test,
3. A Turbine Oil Stability Test, and
4. An Oxygen Absorption Test.

Each of these tests is a modification of a corresponding older test method which has found favor in the examination of uninhibited oils. Two compounded, inhibited oils have been tested by the cooperating laboratories. Each type of test has its advocates but no final conclusions have been drawn pending continuation of the study.

This work will be continued with new inhibited oils, using for further study that test or those tests which have given the greatest promise in the preliminary study described.

Extensive revisions were made in the Standard Method of Test for Power Factor and Dielectric Constant of Electrical Insulating Oils of Petroleum Origin D 924 – 49. These have been approved by Committee D-9 and will be submitted to the Society for adoption.

A new tentative method for the determination of the insulation resistivity of electrical insulating oils of petroleum origin has been prepared and approved by letter ballot of Subcommittee IV. It now will be submitted to Committee D-9 letter ballot for approval to be established as a new tentative under its own ASTM designation.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 49, p. 1041 (1949).

The statements on definition and significance of Power Factor and Resistivity tests, as appearing in Method D 117 - 50 T, have been revised and will appear in the method when reprinted.

Development of the method for determining the dielectric strength of oils of high viscosity is under way and during the coming year the necessary modifications to Method D 877 - 49 to make it suitable for this purpose will be incorporated.

Considerable difficulty is still being experienced in securing satisfactory check tests by different operators in the quantitative determination of water in insulating oils by the Karl Fischer colorimetric and electrometric method. Work is still under way in the refinement of the methods with the hope that one or both methods may be written in sufficient meticulous detail to insure reasonable check test results in different laboratories.

A revision of the sampling procedure for askarels, included in Method D 901 - 50 T, will be submitted to Subcommittee IV for letter ballot approval. If so approved, it will be submitted to Committee D-9 for letter ballot approval as a revision of the tentative method.

Development of a satisfactory quantitative test to determine the amount of sulfide deposited on a copper strip in a proposed new Corrosive Sulfur Test for petroleum electrical insulating oils seems to have been completed. Work during the year, however, developed the fact that apparently the proposed Corrosive Sulfur Test will form slightly variable amounts of sulfide on the copper strip from identical oil samples. Further work is needed to insure uniformity of the test in this respect before a tentative test method can be submitted.

During the year Method D 971 - 49 for determination of "Interfacial Tension of Oil" has been investigated to determine the effect on test results by filtering the sample prior to testing.

Data from four laboratories indicated that on unfiltered oils in the range of 13.0 to 28.0 dynes, filtering the sample increased the readings from 1.0 to 2.0 dynes while those in the range of 5.0 to 9.0 dynes increased 4.0 to 6.0 dynes.

It was the opinion of the subcommittee that, while the interfacial tension test is an excellent tool, it should be used with other tests when judging the serviceability of an oil and that the decision whether or not to filter be left to the individual laboratories.

The Standing Committee of the International Electrotechnical Commission's Committee No. 10, referred to in last year's report, has been active during the year and Subcommittee IV has been kept advised of the progress of its work by its representative, F. M. Clark. A meeting of this International Standing Committee will be held, probably in Sweden, this August and Subcommittee IV recommended unanimously to Committee D-9 that Mr. Clark again be appointed as official representative of Committee D-9 to attend this meeting to cooperate in the work of producing an international method of test to determine the oxidation stability of electrical insulating oils.

A symposium on the results of the subcommittee's cooperative study on the correlation of laboratory tests with service life of uninhibited petroleum electrical insulating oils will be held this fall as noted earlier in this report. Another symposium on the subject of "Reclaiming and Inhibiting Used Electrical Insulating Oils" is planned for the 1952 spring meeting. The speaker's main paper will be prepared well in advance of the meeting and submitted to the membership of Subcommittee IV with a request that members prepare their discussions of the paper in advance of the meeting. It is hoped that many controversial issues on this subject will thus be brought out for free and full discussion.

Brief summaries of the work of Sections A and D of Subcommittee IV are included herein:

Section A on Neutralization Value (R. J. Harrington, chairman) coordinates its efforts with those of Research Division VI, Section A on Neutralization Value, of Committee D-2. A survey was conducted, comparing two color indicator methods, the Proposed Method of Test for Saponification Number of Petroleum Products (Para-Xylenol Blue Color-Indicator Method) and alpha-naphthol benzein (Method D 974), with the electrometric method (Method D 664). Three oil samples were tested and the Tentative Method of Test for Neutralization Value (Acid and Base Numbers) by Color Indicator Titration (D 974 – 48 T) showed the best reproducibility for total acid number. However, it has been pointed out that Method D 974 cannot be used for measuring the total base number. It was proposed that a note be placed in the method stating that the method does not measure total base number and, in such cases, the Standard Method of Test for Neutralization Value (Acid and Base Numbers) by Electrometric Titration (D 664 – 44) should be used. It was voted to continue Method D 974 as a tentative. Committee D-2 will continue its work in the development of a more suitable color indicator method and D-9 Subcommittee IV, Section A, members will cooperate.

Section D on Saponification Number (R. J. Harrington, chairman) received a report on the activities of Committee D-2, Research Division VI, Section A on Saponification Number. A series of round-robin tests were conducted on six oil samples, comparing two colorimetric methods, The Proposed Method of Test for Saponification Number of Petroleum Products (Para-Xylenol Blue Color Indicator Method) and the Tentative Method of Test for Saponification Number of Petroleum Products by Color-Indicator Titration (D 94 – 48 T), with the Standard Method of Test for Saponification Number of Petroleum Products by Potentiometric Titration (D 939 – 50). The para-xylenol blue method appeared promising as a possible substitute for Method D 94 because it is more versatile and is a completely single phase method. The members of the section felt that other ASTM members should have the opportunity to study the para-xylenol blue method which is being recommended by Committee D-2 for publication as information. It was voted to continue Method D 94 as tentative.

Respectfully submitted on behalf of the subcommittee,

E. A. Snyder,
Chairman.

REPORT OF COMMITTEE D-10
ON
SHIPPING CONTAINERS*

Committee D-10 on Shipping Containers held a fall meeting in Philadelphia, Pa., on October 13, 1950, with an attendance of 42, and a spring meeting in Atlantic City, N. J., on April 27, 1951, with an attendance of 56. The fall meeting was followed by an informative talk on "The Nature of Shock and Difficulties in Its Measurement" by Frederic D. Schottland and closed with a luncheon and inspection trip through the courtesy of The Crown Can Co. of Philadelphia.

The spring meeting included an illustrated lecture by Dr. Julian H. Toulouse entitled "A Statistical Inquiry Into Bursting Strength Testing."

The committee received approval from the Board of Directors to enlarge its scope and the revised wording in scope has been accepted and is noted as follows:

> *Scope.*—Nomenclature, definitions of terms, test methods, performance specifications and study of the effect of various factors influencing strength and serviceability relating to packaging, including packages, shipping containers and pallets.

The committee is recommending that a joint committee be set up to coordinate the work of ASTM Committee D-10 and the Container Testing Committee of T.A.P.P.I. looking toward the standardization of test methods. Messrs. Stivers, Bergstrom, and Grundy have been nominated as the representatives of Committee D-10.

Committee D-10 has attracted many capable men and is, without a doubt, now the leading organization in the shipping container field. The meetings are well attended by outstanding individuals from many fields who unselfishly contribute their time and efforts to further the methods of testing and evaluating shipping containers and packages. The present membership consists of 86 members, of whom 30 are classified as producers, 36 as consumers, and 20 as general interest members.

PROPOSED METHOD TO BE PUBLISHED AS INFORMATION

Committee D-10 recommends that the Proposed Methods of Tests for Pallets be published as information, as appended hereto.[1]

ADOPTION OF TENTATIVES AS STANDARD

The committee recommends that the following four tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Methods of Test for:

Water-Vapor Permeability of Packages (D 895 - 47 T),[2]

Water Resistance of Containers by the Spray Method (D 951 - 47 T),[2]

Penetration of Liquids into Submerged Containers (D 998 - 48 T),[2] and

Water Vapor Permeability of Shipping Containers (D 1008 - 49 T).[2]

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* Presented at the Fifty-fourth Annual Meeting of the Society, June 18-22, 1951.

[1] See Editorial Note, p. 387.

[2] 1949 Book of ASTM Standards, Part 4.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends that the following two tentatives be continued in their present status without revision:

Tentative Methods of:

Vibration Test for Shipping Containers (D 999 – 48 T), and

Testing Large Shipping Cases and Crates (D 1083 – 50 T).

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Definitions of Terms (Edward Dahill, chairman) is studying the "Glossary of Terms—Interior Packing" submitted by Subcommittee VI.

Subcommittee II on Methods of Testing (G. E. Falkenau, chairman) is currently studying the vibration test and working on a multiwall bag puncture test.

Subcommittee III on Moisture and Water Vapor Resistance (H. A. Bergstrom, chairman) has proposed investigation of two new cycle tests for water vapor permeability of packages and shipping containers.

Subcommittee IV on Performance Standards (J. H. Toulouse, chairman) voted to recognize four types of handling: domestic, car, truck, or plane load; domestic, less than car, truck or plane load; export, commercial; and military. The National Safe Transit tests are being reviewed with the purpose of possible adoption by Committee D-10 as performance specifications.

Subcommittee V on Correlation of Tests and Test Results (R. C. McKee, chairman) has completed round-robin tests on corrugated boxes in the 7-ft drum test and another series of tests on corrugated boxes in the compression test. It is instigating a series of drop tests and will follow with a series of tests on the incline impact tester. The committee is attempting to discover possible correlation between various laboratories and various test methods.

Subcommittee VI on Interior Packing (S. L. Swenson, chairman) is making a study of possible test methods for evaluting cushioning materials, including shock absorption, abrasiveness and surface protection.

This report has been submitted to letter ballot of the committee, which consists of 91 members; 61 members returned their ballots, of whom 56 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

T. A. CARLSON
Chairman.

EARL R. STIVERS,
Secretary.

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[3] The letter ballot vote of the committee was favorable; the results of the vote are on record at ASTM Headquarters.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-10 presented to the Society through the Administrative Committee on Standards the recommendation that Methods of Test for Pallets be published as tentative. This recommendation was accepted by the Standards Committee on September 12, 1951, and the tentative methods appear in the 1951 Supplement to Book of ASTM Standards, Part 4, bearing the designation D 1815 – 51 T.

REPORT OF COMMITTEE D-11

ON

RUBBER AND RUBBER-LIKE MATERIALS*

Committee D-11 on Rubber and Rubber-Like Materials and fourteen of its subcommittees and sections held meetings over a three-day period in Cincinnati, Ohio, March 7 to 9 during ASTM Committee Week. A number of the subcommittees held separate meetings during the year and all have been actively carrying on their work.

During the week of October 16 to 20, 1950, Committee D-11 was host to ISO Technical Committee 45 on Rubber which met in Akron, Ohio, and actively participated in the ten technical sessions of the international group on rubber standardization. The meetings were presided over by W. J. Naunton of the British Imperial Chemical Industries and were attended by distinguished representatives of seven member countries. Substantial progress in international standardization of rubber testing was made, as evidenced by agreement on 112 resolutions covering a wide range of subjects. A highlight of the meeting was a complimentary banquet for the delegates on the evening of October 17, provided by the American rubber companies. The next meeting of ISO/TC/45 will be held in England in October, 1951.

At the time of the ISO/TC/45 meeting in Akron, advantage was taken by the D-11 Subcommittees on Crude Rubber and Latex of the presence of a number of technical representatives from the rubber-producing areas in the Far East to obtain their valuable assistance. Both subcommittees held meetings in Akron at which the many problems connected with the sampling, grading, and testing of these materials were thoroughly discussed. One particularly interesting development is the production in Indo-China of a series of specially controlled crude rubbers which are technically graded according to viscosity and rate of cure. These rubbers are now available and are being investigated further by the committee. As reported last year, the work on standard reference samples of rubber compositions has been retarded by the difficulty in obtaining an adequate supply of a uniform standard crude rubber. This is still delaying adoption of standard rubber compound formulations. However, work is continuing on this problem, and the close relations which have now been established with the crude rubber producers, together with the work which they are doing on technical grading and production of specification rubbers, may lead to the early establishment of the desired standards.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1950 Annual Meeting, Committee D-11 presented to the Society through the Administrative Committee on Standards the following recommendations:

Tentative Methods of Test for:

Compressibility and Recovery of Gasket Materials (D 1147 – 51 T),

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* Presented at the Fifty-fourth Annual Meeting of the Society, June 18–22, 1951.

Discoloration of Vulcanized Rubber: Organic Finish Coated or Light Colored (D 1148 - 51 T), and
Accelerated Ozone Cracking of Vulcanized Rubber (D 1149 - 51 T).

Revision of Tentative Methods of:

Testing Flat Rubber Belting (D 378 - 50 T),
Testing Rubber Hose (D 380 - 50 T),
Dynamic Testing for Ply Separation and Cracking of Rubber Products (D 430 - 49 T),
Test for Changes in Properties of Rubber and Rubber-Like Materials in Liquids (D 471 - 49 T),
Testing Automotive Hydraulic Brake Hose (D 571 - 48 T),
Testing Asphalt Composition Battery Containers (D 639 - 49 T), and
Test for Contact and Migration Stain of Vulcanized Rubber in Contact with Organic Finishes (D 925 - 47 T).

Revision of Tentative Specifications and Methods of Test for:

Sponge and Expanded Cellular Rubber Products (D 1056 - 49 T).

These recommendations were accepted by the Standards Committee on May 11, 1951.

On May 15, 1951, the Standards Committee accepted the revision of the Tentative Specifications for Rubber and Synthetic Rubber Compounds for Automotive and Aeronautical Applications (D 735 - 48 T).

On May 26, 1951, the following two recommendations were accepted by the Administrative Committee on Standards:

Emergency Alternate Revisions of Tentative Specifications for:

Friction Tape for General Use for Electrical Purposes (D 69 - 48 T), and
Rubber Insulating Tape (D 119 - 48 T).

All of the new and revised methods and specifications will be published in the 1951 compilation of "ASTM Standards on Rubber Products."

The new Tentative Method of Test for Compressibility and Recovery of Gasket Materials provides the means for evaluating important characteristics of these compounds. It is quite broadly applicable to automotive gaskets and is a definite improvement over the compressibility test already specified in the Methods of Testing Compressed Asbestos Sheet Packing (D 733 - 50), which the committee plans to revise at an early date. The Methods of Test for Discoloration of Vulcanized Rubber: Organic Finish Coated or Light Colored were developed by the SAE-ASTM Technical Committee on Automotive Rubber to fill a need for a means of evaluating the effect of heat and sunlight on white or light-colored vulcanized rubber which has been covered with an organic finish. The new Tentative Method of Test for Accelerated Ozone Cracking of Vulcanized Rubber was originally proposed by the Bell Telephone Laboratories as a method for measuring in the laboratory the tendency of different grades of rubber to develop ozone cracking when exposed under strain to the action of the atmosphere, as in automotive uses. This type of cracking has long been recognized, but is exceedingly difficult to evaluate because it is apparently due to the very small concentrations of ozone normally present in atmospheric air. Correlation and duplication of results under these conditions is difficult, but this method has been found to be the best available at present.

The revision of the Tentative Methods of Testing Flat Rubber Belting (D 378) is quite extensive and incorporates changes in test procedures previously adopted in the Tension Test Methods D 412. In this revision the belting methods are brought up to date with respect to the more recent developments in the synthetic rubber program. The revision of the Rubber Hose Testing Methods (D 380) adds procedures, not previously included, for testing tube and cover adhesion for hose containing woven-wire reinforcement. The change in the Dynamic Test for Ply Separation and Cracking (D 430) has involved a complete rewriting of method C of the tentative which covers the use of the duPont flexing machine. A number of inaccuracies are corrected and the procedure is brought in line with present practice. The revision of the Immersion Test Methods (D 471) improves their usefulness by adding a durometer hardness test and by providing for the use of an air condenser to prevent losses from oils when testing at the higher temperatures. In the Methods of Testing Automotive Hydraulic Brake Hose (D 571) changes have been made to include procedures for testing an additional size of hose, namely, $\frac{1}{4}$-in. hose which is now being widely used and has not previously been covered. In the Methods of Testing Asphalt Battery Containers (D 639), the procedures for conducting the tests for acid absorption and for bulging due to service-generated heat have been changed and improved. In the Methods of Test for Contact and Migration Stain of Rubber in Contact with Organic Finishes (D 925) the revision adds an alternate light source for method B on migration stain which is in considerable use and has been found satisfactory.

The revision of the Tentative Specification and Methods of Test for Sponge and Expanded Cellular Rubber Products (D 1056) provides an additional grade of closed-cell expanded rubber in each of the three tables of physical properties in these specifications. Experience has indicated a need for these additional grades.

The SAE-ASTM Technical Committee on Automotive Rubber has worked for several years on a revision of the Tentative Specification for Rubber and Synthetic Rubber Compounds (D 735). Increased availability of crude rubber, changes and improvements in the synthetic rubbers, as well as increased experience with the use of these specifications, have all indicated a need for revision and simplification. This task has involved, principally, a complete review of the specifications, changes in the basic requirements of the various grades to include oven aging and compression set limitations, and a consolidation of the two type R tables which have no requirement for oil resistance. This work was completed and agreement reached in the technical committee prior to the outbreak of the Korean War. Before final approval in Committee D-11 was obtained, the national defense situation and the imposition of rubber controls by Government regulation raised the question as to whether the newly revised specifications would be suitable during the period of the new emergency. This matter was thoroughly considered by Committee D-11 at Cincinnati and by the Automotive Technical Committee, with the final decision that the revised specifications should be issued, even though difficulty might be experienced in a few cases by rubber manufacturers in meeting certain of the physical requirements specified in the new type R table. It is believed that, with some experience under the new Government regulations, most of these difficulties will be overcome and, meanwhile, any deviations which may be necessary can be satisfactorily arranged by agreement between the parties concerned. The revised specifications contain a note to this effect.

EMERGENCY ALTERNATE PROVISIONS

The Government restrictions referred to created a somewhat more serious situation in the case of the Tentative Specifications for Friction Tape (D 69) and for Rubber Insulating Tape (D 119). In both of these specifications the reduced allocations of crude rubber led to the recommendation by the committee to the Administrative Committee on Standards of the publication of emergency alternate provisions as referred to earlier in this report. In specifications D 69 the adhesion test after oven aging is omitted and the permissible number of pin holes is slightly increased. In D 119 the requirement for natural rubber was eliminated and the limitations on tensile strength, elongation, and dielectric strength were somewhat modified.

NEW TENTATIVES

Committee D-11 recommends the acceptance for publication of the following two new tentatives as appended hereto:[1]

Tentative Specifications for Gasket Materials for General Automotive and Aeronautical Purposes.—These specifications have been developed by Section X on Automotive Gaskets (J. P. Wilson, chairman) of the SAE-ASTM Technical Committee on Automotive Rubber. They have been prepared for use in selecting gasket materials for general automotive and aeronautical applications. These specifications are comprehensive in that they cover gasket materials composed of natural rubber, synthetic rubber, cork, treated and untreated papers, asbestos, and various combinations of these materials. Work on these specifications has been in progress for over two years, and they represent a wealth of information based on experience with gasket materials in a wide variety of automotive and industrial applications.

Tentative Method of Test for Weather Resistance Exposure of Automotive Rubber Compounds.—This method has been developed by the SAE-ASTM Technical Committee on Automotive Rubber after considerable study and comparative testing. The method will provide a very useful standard for evaluation of automotive rubber compounds.

REVISIONS OF TENTATIVES

The committee recommends that three of the present tentatives be revised as indicated and continued as tentative:

Tentative Method of Test for Tension Testing of Vulcanized Rubber (**D 412 - 49 T**).[2]—This revision provides for the use of a greater rate of jaw separation speed than the standard of 20 in. per min used in routine testing, providing that a statement of the actual speed used is included with the results. This does not change the standard speed in cases of arbitration but will permit more efficient routine and control testing. Investigations by the subcommittee have indicated that the increase within the limits specified affects the results obtained to a minor degree in most cases. The proposed revision is as follows:

Section 3(e).—Immediately after the third sentence which reads: "The rate of travel of the power-actuated grip shall be 20 ±1 in. per min and shall be uniform at all times," add the following sentence: "If conditions allow and a higher rate of separation is desirable, an increase up to a 40-in. rate may be used in routine work and notation of the speed used made on the report, but in case of a dispute, the 20-in. rate shall be considered standard."

Tentative Methods of Testing Rubber Adhesives (**D 816 - 46 T**).[2]—The following change is recommended in order to make the test methods more definite and to eliminate questions which have arisen concerning the requirement: In Sections 7 (*a*) and (*b*), 10, 12 and 27, revise the phrase "as near as possible 0.001 in. in thickness" to read "0.001 in. in thickness or of an optimum thickness suitable to the product, as agreed by the parties concerned."

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[1] The new tentatives were accepted by the Society and appear in the 1951 Supplement to Book of ASTM Standards, Part 6.

[2] 1949 Book of ASTM Standards, Part 6.

Tentative Method of Measuring Low-Temperature Stiffening of Rubber and Rubber-Like Materials by the Gehman Torsional Apparatus (**D 1053 - 49 T**).[2]—This revision provides for more accurate standardization of the torsion wire used in the apparatus against which the test specimen is evaluated. Other changes include more suitable dimensions for the test specimen and definite instruction for positioning the thermocouple.

Section 2 (*c*).—Revise the first sentence to read as follows: "Torsion apparatus for measuring the stiffening of the test specimens shall consist of a torsion head, *H*, and a torsion wire, *I*, of tempered spring wire 2.5 ± 0.2 in. long having a torsional constant of 0.500 g-cm per degree of twist."

Section 3.—Revise the second sentence, giving the dimensions of the specimens, to read as follows: "Small rubber strips 1.5 ± 0.1 in. long, 0.125 ± 0.005 in. wide, and 0.077 ± 0.005 in. thick are suitable for this test."

Section 4 (*b*).—Revise the sentences in parenthesis to read as follows: "(If the reading at 25 C does not fall in the range of 170 to 120 deg, the standard torsion wire is not suitable for testing the specimen. Specimens twisting more than 170 deg shall be tested with a wire having a torsional constant of 0.125 g-cm per degree of twist. Specimens twisting less than 120 deg shall be tested with a wire having a torsional constant of 2.000 g-cm per degree of twist.)"

Section 4 (*d*).—Revise the sixth sentence to read as follows: "The thermocouple shall be inserted in the chamber witb the junction halfway between the top and the bottom of the specimen and the air flow shall be started and continued until the temperature in the chamber has dropped to the lowest test temperature desired."

REVISION OF STANDARDS AND REVERSION TO TENTATIVE

Standard Method of Test for Indentation of Rubber by Means of the Pusey and Jones Plastometer (**D 531 - 49**),[2] *Standard Methods of Testing Compressed Asbestos Sheet Packing* (**D 733 - 49**).[2]—Revision of these standard methods is in progress and it is expected that they will be presented to the Society through the Administrative Committee on Standards prior to the next Annual Meeting. In the case of the Pusey and Jones Plastometer, a newly designed instrument has become available which is expected to introduce extensive changes in both the equipment and method for this test. In the asbestos sheet packing methods, the compressibility test is being revised to conform with the newly adopted tentative method for compressibility and recovery (D 1147). The committee requests that these standards be reverted to tentative status to facilitate this work.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends that all tentatives under its jurisdiction, other than those mentioned in this report, be continued as tentative. Most of these are being reviewed by the various subcommittees and the results of this work will be presented to the Society as it is completed.

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

ACTIVITIES OF SUBCOMMITTEES

Committee D-11 has authorized formation of a new subcommittee to develop specifications for rubber compounds for general application similar to the SAE-ASTM Specifications D 735, which have been used so successfully in the automotive and aeronautical fields. This task will require extended study by the new subcommittee in order to cover adequately the broad industrial field, but it is believed that such specifications, when completed, will find many applications. The new Subcommittee XXVIII on the Application of Statistical Quality Control Methods to Rubber Products, previously authorized, is now in process of organization. Mr. J. D. Heide of the U. S. Rubber Co. has been appointed chairman. Resignations made necessary by change in duties of M. G. Schoch, Jr., chairman of Subcommittee II on Belting, and R. M. Howlett, chairman of Subcommittee XIX on Immersion Tests, have been accepted with regret. Mr. W. N. Keen has been appointed to replace Mr. Howlett but no replacement has yet been designated for Mr. Schoch.

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[3] The letter ballot vote of the committee was favorable; the results of the vote are on record at ASTM headquarters.

The various subcommittees of Committee D-11 have been exceedingly active during the year. The preparation of a new specification for electrical gloves by Subcommittee IV was mentioned last year. This work is now practically complete. Subcommittee XV has a Tentative Method of Test for Resistance to Aging of Vulcanized Rubber by Measurement of Creep in draft form, and Subcommittee XVII is considering a draft of a Tentative Method for Determining Compression Set at Low Temperatures. Subcommittee XXI expects to complete its consideration of Tentative Methods of Test for Rubber Brake Bonding Materials at this Annual Meeting. These new tentatives will probably be ready for presentation to the Society for publication within the next few months.

Other tentatives which are being considered include: Proposed Specifications for Polyethylene and Butyl Rubber Insulated Power Cables, in Subcommittee V; Specifications for Ozone Resisting Tape, in Subcommittee IX; and a Procedure for Determining Specific Gravity and Volume Increase, using the Jolly Balance, in Subcommittee XIX. Subcommittee X is progressing in its work of revision of Methods D 15, covering sample preparation for physical testing of rubber products. This has proven to be a more difficult task than anticipated and the methods are being completely reorganized and rewritten.

Many other interesting activities are under way. The cooperative test program in Subcommittee XV on correlation of oven and shelf aging has now yielded results covering a period of three years. Subcommittee XVII is studying the phenomenon of stress relaxation in rubber and is planning a round-robin test on four different gasket compounds.

Subcommittee XIX has been notified that the commercial source of ASTM reference fuel No. 2 used in immersion tests is being discontinued, and it is investigating the possible substitution of *iso*-octane or similar fluids. Subcommittee XX has been requested by the ISO Technical Committee on Rubber to investigate the effect of temperature on ply adhesion and the effect of humidity on the adhesion when the articles contain synthetic textiles.

Two very interesting papers on forced vibration test methods were presented before Subcommittee XXVII on Resilience at its spring meeting. These were: "The Measurement of Hysteresis and Dynamic Modulus of Elastomers by a Vector Subtraction Method,"[4] by G. W. Painter of the Lord Manufacturing Co., and "The Buick Motor Mount Testing Machine," by Lloyd Muller, Buick Motor Division, General Motors Corp. This subcommittee is working on the development of a Tentative Method of Test for Resilience using Forced Vibration.

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[4] Published in the ASTM BULLETIN, No. 177, October, p. 45 (TP 209).

From the above summary, it is obvious that Committee D-11 and its various subcommittees are vigorously at work in the field of rubber standardization.

This report has been submitted to letter ballot of the committee, which consists of 198 members; 104 members returned their ballots, of whom 97 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

SIMON COLLIER,
Chairman.

ARTHUR W. CARPENTER,
Secretary.

REPORT OF COMMITTEE D-12

ON

SOAPS AND OTHER DETERGENTS*

Committee D-12 on Soaps and Other Detergents held one meeting during the year on March 19 and 20, 1951, in New York City.

Mr. F. W. Smither has been elected honorary chairman of Committee D-12.

NEW TENTATIVES

The committee recommends that the following methods of test be accepted by the Society for publication as tentative:

Tentative Methods of Test for:

pH of Aqueous Solutions of Soaps and Detergents,[1] and

Foaming Properties of Surface-Active Agents.[2]

REVISIONS OF TENTATIVES

The committee recommends that the following specifications be revised as indicated:

Tentative Specifications for:

Chip or Granular Soap for Low-Temperature Washing (Low and Medium Titer) (D 1111 - 50 T)[3] as appended hereto.[4] The revision consists of: the inclusion of requirements for a new Type C; changing the value in Table I for "Sum of free alkali or free acid, total matter insoluble in alcohol, and sodium chloride, per cent" from "4.0" to "6.0" for both Types A and B; and rewording the introduction of Footnote *b* under Table I to read "Acceptable inhibitors, of the following type, shall be used, unless otherwise specified:".

Solid Soap for Low-Temperature Washing (Low and Medium Titer) (D 1112 - 50 T),[3] revised by: changing in Table I the requirements for "Sum of free alkali or free acid, total matter insoluble in alcohol, and sodium chloride, per cent" from "3.0" to "4.0" for both Types A and B; and by rewording the introduction of Footnote *b* under Table I to read "Acceptable inhibitors, of the following type, shall be used, unless otherwise specified:".

REVISION OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption the following revisions in four standards and accordingly requests the necessary nine-tenths affirmative vote at the Annual Meeting in order that these recommendations may be referred to letter ballot of the Society:

Standard Specifications for Chip Soap (**D 496 - 39**),[5] and *Standard Specifications for Powdered Soap (Nonalkaline Soap Powder)* (**D 498 - 39**).[5]—Revise to provide for an increase in the permissible value for "sum of free alkali, total matter insoluble in alcohol, and sodium chloride, max., per cent" from 4.0 to 6.0.

Standard Specifications for Liquid Toilet Soap (**D 799 - 45**).[5]—Revise the maximum permissible amount of matter insoluble in alcohol by changing from "0.4 per cent" to "0.5 per cent."

Standard Definition of Terms Relating to Soaps and Other Detergents (**D 459 - 48**).[5]—The committee recommends revision to include the following additional definitions:

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* Presented at the Fifty-fourth Annual Meeting of the Society, June 18–22, 1951.

[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 50, p. 362, (1950).

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 50, p. 363, (1950).

[3] 1950 Supplement to Book of ASTM Standards, Part 5.

[4] The revised tentative was accepted by the Society and appears in the 1951 Supplement to Book of ASTM Standards, Part 5.

[5] **1949 Book of ASTM Standards, Part 5.**

Lather.—A foam or froth when a detergent is agitated in water or other liquid.

Foam.—A mass of bubbles formed on liquids by agitation.

Suds.—A foam or lather generated on or in a detergent solution.

Rinse (whether used as a verb or a noun).—A treatment with water or other solvent for the purpose of removing dirt or detergent.

Emulsion.—A suspension of fine particles or globules of one or more liquids in another liquid.

Emulsion Cleaner.—A composition which forms an emulsion capable of dissolving or suspending soil.

Soil (in reference to detergency).—A matter out of place.

Diphase Metal Cleaner.—A composition which produces two phases in the cleaning tank, namely, a solvent layer and an aqueous layer, which cleans by solvent action and emulsification.

TENTATIVE CONTINUED WITHOUT REVISION

The committee recommends that the Tentative Specifications for Sodium Bicarbonate (D 928 - 47 T) be continued without revision.

EDITORIAL CHANGE IN STANDARD

The committee recommends that the following methods be editorially revised as indicated:

Standard Methods of Sampling and Chemical Analysis of Soap and Soap Products (**D 460 - 46**).[5]—Revise the last sentence of Section 21 (*b*), which at present reads "One milliliter of 0.1 *N* $AgNO_3$ solution is equivalent to 0.00585 g of NaCl," to read "One milliliter of the 0.1 *N* $AgNO_3$ solution is equivalent to 0.00585 g of NaCl or 0.00746 g of KCl."

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[6]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee T-1 on Soap Analysis (E. W. Blank, chairman).—In this joint committee of the American Oil Chemists' Society and the American Society for Testing Materials, cooperative work on the development of a method for the determination of water insoluble material in soaps is being continued, and a similar investigation of a method for the determination of borax in soap is to be undertaken. The committee has under advisement the use of methyl purple indicator as an alternate to methyl orange in soap analysis. Methods for the determination of copper in soap are under study.

Subcommittee T-4 on Analysis of Alkaline Detergents (W. A. Koch, chairman) has a method for analysis of sodium bicarbonate which is considered satisfactory for referee purposes but will give further consideration to methods suitable for routine use before making any recommendations.

Subcommittee T-5 on Physical Testing (J. A. Woodhead, chairman) has prepared a proposed method of test for surface and interfacial tension of solutions of surface-active agents, and intends to recommend its publication as information. A symposium was held on methods for the determination of wetting-out properties of surface active agents.[7] The committee will continue work on this subject with the aim of establishment of standard methods.

Subcommittee T-6 on Metal Cleaning (J. C. Harris, chairman).—Subcommittee T-6 has completed the preparation of proposed methods for the evaluation of rinsing and buffering properties of metal cleaners, and a total immersion corrosion test for soak tank metal cleaners, and intends to recommend the publication of these methods as information. Preparation of a procedure for the evaluation of emulsification properties of detergents is to be continued. The committee plans to undertake work in setting up a recommended outline for soil removal procedures for the evaluation of metal cleaners. The committee plans to investigate the subject of corrosion in washing machines.

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[6] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM headquarters.

[7] Published in Special Technical Publication *STP No. 115*, January, 1952.

Subcommittee S-1 on Specifications for Soap (Frederick Krassner, chairman).—The subcommittee recommended revision of tentative specifications for solid, chip and granular soap for low-temperature washing, as mentioned earlier in this report. The subcommittee is cooperating with the Federal Specifications Board in the interest of coordination. During the coming year the subcommittee intends to make further studies of specifications for concentrated liquid soaps.

The committee has accumulated technical information from producers of sodium carboxymethyl cellulose and will turn this over to the Committee on Chemical Cellulose which is in the progress of organization. After preliminary studies the adoption of specifications for trisodium phosphate monohydrate has been deferred because of lack of interest, and specifications for pentasodium tripolyphosphate has been deferred for lack of adequate methods of test. The committee plans to take up work on specifications for sodium orthosilicate and for caustic potash.

Subcommittee G-4 on Program Planning (R. B. Mitchell, chairman).—This committee, organized during the past year, made arrangements for the 1951 meeting which included the presentation of eight technical papers and reports on the activities of the following organizations with respect to testing and specifications for detergents: National Security Industrial Assn; Chemical Specialties Manufacturers' Assn; and American Assn of Textile Chemists and Colorists.

The following papers were presented:

"An In-vivo Method for Determining the Degerming Powers of Soap Containing Hexachlorophene," by Dr. Cade,[7]

"Practical Evaluation of Soiled Test Pieces," by R. B. Mitchell,[7]

"A Method for Measuring the Adsorption of Anion-Active Agents on Materials Commonly Washed," by W. A. Fessler,[7]

"The Mechanical Effect Produced in Launderometer Jars," by O. C. Bacon,

"The Mechanism of the Wetting of Textile," by Irving Gruntfest,[7]

"A Laboratory Performance Test for Detergents Used in Continuous Wool Scouring," by E. A. Leonard,[7]

"Function of Soaps and Detergents in Dry Cleaning," by G. P. Fulton, and

"A Metal Cleaning Test Using Radioactive Stearic Acid as Soil," by J. W. Hensley, H. A. Skinner, and H. R. Suter.[7]

This report has been submitted to letter ballot of the committee, which consists of 150 members; 76 members have returned their ballots, all of whom have voted affirmatively negatively.

Respectfully submitted on behalf of the committee,

J. C. HARRIS,
Chairman.

H. R. SUTER,
Secretary.

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[7] Published in Special Technical Publication *STP No. 115*, January, 1952.

REPORT OF COMMITTEE D-13

ON

TEXTILE MATERIALS*

Committee D-13 on Textile Materials held two 3-day meetings during the year, both of them in New York City. The time usually given to a papers session was devoted to meetings of task groups. The reaction to this innovation was generally favorable since task groups have found it difficult to meet independently.

Papers on the following subjects were presented at meetings of the subcommittees during the year:

Subcommittee A-1, Section I:

"Recent Developments on the Uses of the Arealometer," by K. L. Hertel, University of Tennessee, (March, 1951 Meeting)

Subcommittee A-10, Section I:

"Resistance of Wool and Wool Felt to Heat," by C. C. Gordon, Albany Felt Co. (March, 1951)

Subcommittee B-1:

"Application of the Specific Gravity Gradient Columns to the Detection of Additives in Textiles," by C. R. Stock, American Cyanamid Co. (October, 1950)

"International Standards for Testing Yarns," by J. K. Frederick, Jr., Lowell Textile Inst. (October, 1950)

"A Study of Fabric Bending Methods of P. T. Peirce," by N. J. Abbott, Ontario Research Foundation.[1] (March, 1951)

"The Relation of Fiber Properties to the Hand of Fabrics," by L. G. Ray, E. I. duPont de-Nemours & Co.[1] (March, 1951)

Subcommittee B-5:

"Cotton Backing Yarn Specifications, Their Development and Use," by C. W. Carter and R. G. Levitch, Bigelow-Sanford Carpet Co. (October, 1950)

"Quick Statistical Analysis of Data," by E. Rupert Gause, Bell Telephone Laboratories. (March, 1951)

An important change in organization of Committee D-13 was made this year. Two subcommittees which dealt chiefly with nonwoven fabrics, Subcommittee A-10 on Felt and Subcommittee A-14 on Bonded Fabrics, have been combined to form Subcommittee A-10 on Non-Woven Fabrics with R. R. Stevens as chairman, G. B. Harvey, vice-chairman, and K. H. Barnard, secretary. The new subcommittee has two sections: Section I on Felt with R. R. Stevens as chairman, and R. H. Carter as secretary; and Section II on Bonded Fabrics with G. B. Harvey as chairman and K. H. Barnard as secretary.

The following changes in subcommittee and section chairmanships were approved by the Advisory Committee during the year: G. S. Buck, Jr., vice R. L. Lee, Jr., as chairman of A-1, Section I; G. B. Krumholz, vice K. N. Mathes, as chairman of Subcommittee A-8, Section III.

At the fall meeting a luncheon was held in honor of the two charter members of Committee D-13, Daniel E. Douty and Horace J. Jaquith. Each spoke on his experiences in the committee since its organization in 1914, and each was given a scroll expressing appreciation of their loyalty and interest in the work over the years.

The second meeting of ISO Technical

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* Presented at the Fifty-fourth Annual Meeting of the Society, June 18–22, 1951.

[1] ASTM BULLETIN, No. 176, September, 1951, p. 46 (TP 184).

Committee 38 on Textile Materials is being held in Bournemouth, England, June 4 to 9. The U.S.A. delegation consists of fifteen, nine of them members of Committee D-13. The ASTM is a co-sponsor with the American Association of Textile Chemists and Colorists of the American group dealing with ISO/TC 38 projects, and Committee D-13 is taking an active part in the work. A report on the Bournemouth meeting will be published in the ASTM BULLETIN.[2]

RECOMMENDATIONS AFFECTING STANDARDS

Committee D-13 is presenting for publication 1 new tentative and is recommending the revision of 4 tentatives, tentative revisions in 6 standards, adoption as standard of several definitions and 4 tentatives, immediate revision of 2 standards, the withdrawal of 1 tentative and 1 standard, and editorial changes in 1 tentative and 2 standards.

The standards and tentatives affected, together with the revisions recommended, are given in detail in the Appendix.

All other tentatives not specifically referred to are being actively studied by the respective sponsoring subcommittees.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee A-1 on Cotton and Its Products (B. L. Whittier, chairman):

Section I on Cotton (G. S. Buck, Jr., chairman) is continuing its study of improved methods of test for the various properties of cotton.

Section II on Cotton Yarns and Threads (W. R. Marsden, chairman) is continuing its work on revision of the Methods of Testing and Tolerances for Cotton Yarns (D 180 - 49 T) and for Cotton Sewing Thread (D 204 - 42).

Subcommittee A-2 on Man-Made Organic Base Fibers and Their Products (A. M. Tenney, chairman):

Section I on Spun and Continuous Filament Man-Made Organic Base Yarns (J. K. Frederick, Jr., chairman) is continuing its work on methods of testing these yarns.

Section II on Fabrics (G. H. Hotte, chairman) is working on a new tentative on fabric distortion and revision of the Standard Method of Test for Resistance to Yarn Slippage in Silk, Rayon and Estron Woven Fabrics (D 434 - 42).

Subcommittee A-3 on Wool and Its Products (Werner von Bergen, chairman):

Section I on Wool (O. P. Beckwith, chairman) is continuing its survey of wool testing. It has task groups working on the determination of fineness, moisture, tensile strength, and alkali solubility of wool. It is also working on a revision of the Standard Method of Test for Hard Scoured Wool in Wool in the Grease (D 584 - 50) and on Methods of Core Sampling of Wool in Packages for Determination of Percentage of Hard Scoured Wool Content (D 1060 - 49 T). An interlaboratory check is being made on fiber length testing.

Section III on Woolen and Worsted Yarns (H. R. Anderson, chairman) is working on a revision and consolidation of Specifications and Methods of Test for Woolen Yarns (D 403 - 48 T), for Worsted Yarns (D 404 - 48 T), and for Yarns Spun from Mixed Fibers (D 508 - 43).

Subcommittee A-4 on Asbestos and Its Textile Products (F. S. Mapes, chairman) is working on the preparation of significance of test statements for asbestos textiles, statistical accuracy of test methods, and revision of sampling of asbestos rovings.

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[2] ASTM BULLETIN No. 174, May, 1951, p. 36.

[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee A-5 on Bast and Leaf Fibers and Their Products (S. J. Hayes, chairman) is continuing its studies of test methods for twine. The task group on knot breaking has been reappointed.

Subcommittee A-7 on Ultimate Consumer Textile Products (A. F. Tesi, chairman) is working on methods of testing slide fasteners and procedures for field testing of consumers' textile products. As this subcommittee was organized to develop test methods for finished consumer textile products, it is recommending the withdrawal of Tentative Specifications for Bleached Cotton Broadcloth (D 504 - 41 T) and Standard Specifications for Terry (Turkish) Toweling (D 505 - 40).

Subcommittee A-8 on Glass Fiber and Its Products (F. A. Mennerich, chairman) is continuing its study of methods for determining spread and build-up of glass yarns for wrap and braided covers of wires.

Subcommittee A-9 on Tire Cord and Fabrics (F. S. Kennedy, chairman) is continuing its work on separate revisions of Methods of Tests and Tolerances for Cotton Tire Cord (D 179 - 46 T), and for Rayon Tire Cord (D 885 - 46 T), and on air-actuated clamps.

Subcommittee A-10 on Nonwoven Fabrics (R. R. Stevens, chairman; G. B. Harvey, vice chairman):

Section I (formerly Subcommittee A-10) *on Felt* (R. R. Stevens, chairman) is continuing its work on measurement of stiffness of felts and thickness of cut parts.

Section II (formerly Subcommittee A-14) *on Bonded Fabrics* (G. B. Harvey, chairman) is continuing its work on water absorbency, fire resistance, abrasion resistance, air permeability, laundering and dry cleaning, flexural rigidity, and tear resistance.

Subcommittee A-11 on Pile Fabrics (E. A. Leonard, chairman) is continuing its work on abrasion testing, mothproofness, fire resistance, light-fastness standards, and effect of shampooing on color.

Subcommittee B-1 on Test Methods (W. M. Scott, chairman) is continuing its study of abrasion techniques, standard time for break test, electronic testing machines, dimensions of jaws for grab test, general method of testing yarns, luster, hand of fabrics, internal friction, crease resistance, fraying of fabrics, identification of finishes, copper and manganese in textiles, analysis of mixtures containing vinyon, shrinkage of knit fabrics; also resistance to insect pests, water, and fire.

Subcommittee B-2 on Nomenclature and Definitions (A. G. Scroggie, chairman), besides working on the development of definitions, is continuing the study of certain definitions to bring about a better agreement between ASTM and British definitions.

Subcommittee B-5 on Sampling, Presentation and Interpretation of Data (J. C. Hintermairer, chairman) is continuing Recommended Practice for Planning Interlaboratory Testing of Textile Materials (D 990 - 49 T) as tentative, pending further study of its use. Work has been renewed on sampling part wool blankets for wool content. A short paper on statistical methods is being presented at each meeting.

This report has been submitted to letter ballot of the committee, which consists of 348 members; 163 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

W. D. APPEL,
Chairman.

W. H. WHITCOMB,
Secretary.

APPENDIX

RECOMMENDATIONS AFFECTING STANDARDS FOR TEXTILE MATERIALS

In this Appendix are given proposed revisions in certain standards and tentatives covering textile materials, which are referred to earlier in this report. These standards and tentatives appear in their present form in the 1949 Book of ASTM Standards, Part 5, or in the 1950 Supplement to Book of ASTM Standards, Part 5.

NEW TENTATIVE

Tentative Methods of Test for Resistance to Abrasion of Textile Fabrics:

Committee D-13 recommends these methods, which cover five separate test procedures, for publication as tentative, as appended hereto.[1] These methods were developed after long and careful study of a number of abrasion tests by the Section on Abrasion (C. L. Simon, chairman) of Subcommittee B-1.

REVISION OF TENTATIVES

Tentative Definitions of Terms Relating to Textile Materials (D 123 – 50 T):[2]

The committee recommends that this tentative be revised by the addition of the following definitions of terms: Abrasion; Conformance; Consignment; Elongation; Hygrometer; Psychrometer; Rayon (*adj.*); Shrinkage; Tare; Warp; Weight, Gross; Weight, Net; and Yarn. These new definitions appear in the proposed revision of D 123, as appended hereto.[3]

Tentative Specifications and Methods of Test for Asbestos Yarns (D 299 – 50 T),[2] and

Tentative Specifications and Methods of Test for Asbestos Lap (D 1061 – 50 T):[2]

On the recommendation of Subcommittee A-4, it is proposed that in Section 2 (*e*) of Methods D 299 and in Section 3 of Methods D 1061 which defines "Standard Condition," the humidity requirement be changed from "65 per cent" to "50 per cent."

Tentative Method of Test for Apparent Fluidity of Dispersions of Cellulose Fibers (D 539 – 48 T):[4]

On the recommendation of Subcommittee B-1, the committee recommends that Methods D 539 be revised to include three separate methods as follows: Method A (Sections 2 to 7) is continued with no change; Method B (Sections 8 to 14) is revised as appended hereto[5]; new Method C is being added as appended hereto.[5]

TENTATIVE REVISIONS OF STANDARDS

On the recommendation of Subcommittee A-4, a tentative revision of the following four standards is proposed which provides that in the section indicated, which defines "standard condition," the humidity requirement be changed from "65 per cent" to "50 per cent."

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[1] The new tentative was accepted by the Society and appears in the 1951 Supplement to Book of ASTM Standards, Part 5.

[2] 1950 Supplement to 1949 Book of ASTM Standards, Part 5.

[3] The revised definitions were accepted by the Society and appear in the 1951 Supplement to Book of ASTM Standards, Part 5.

[4] 1949 Book of ASTM Standards, Part 5.

[5] The revised method was accepted by the Society and appears in the 1951 Supplement to Book of ASTM Standards, Part 5.

Standard Specifications and Methods of Tests for Asbestos Tape for Electrical Purposes (D 315 - 49),[4] Section 16,

Standard Specifications and Methods of Tests for Asbestos Roving for Electrical Purposes (D 375 - 49),[4] Section 2 (*c*),

Standard Methods of Testing Woven Asbestos Cloth (D 577 - 42),[4] Section 3, and

Standard Methods of Testing Asbestos Tubular Sleeving (D 628 - 44),[4] Section 3.

Standard Methods of Testing Felt (D 461 - 50):[2]

On the recommendation of Subcommittee A-10, the following proposed change is submitted as a tentative revision of this standard:

Section 8 (*e*).—In Item (*2*) add the following sentence after the third sentence: "The parts to be nested shall be cut so as not to cause excessive compression of the part and shall be conditioned in accordance with Section 3 after cutting."

Standard Methods of Testing and Tolerances for Single Jute Yarn (D 541 - 49):[4]

On the recommendation of Subcommittee A-5, the following proposed change is submitted as a tentative revision of this standard:

Section 10 (*b*).—After the tabulation, insert the following note:

NOTE.—To avoid sampling the same spinning bobbin twice, duplicate skeins from any package shall be taken at least 1 in. apart radially in the thickness of the tube or cone; or, at the option of the tester, two tubes instead of one may be taken at random from each of 10 bales, and one skein reeled from each of 20 tubes; making in all 20 test skeins by either procedure.

ADOPTION OF TENTATIVES AS STANDARD

Tentative Definitions of Terms Relating to Textile Materials (D 123 - 50 T):[2]

The committee recommends that the following definitions now published under D 123 - 50 T be approved for reference to letter ballot of the Society for adoption as standard: Acetate; Acetate, Modified; Extensibility; and Yarn, Kraft.

It is also recommended that the following three terms now appearing in Tentative D 123 be adopted and included in the glossary, which constitutes Appendix I of Standard D 123: Carding, with an editorial change to delete the words "The reduction of" and replace by "The separation and partial cleaning by processing. . ."; Hair; and Noil.

Tentative Specifications for Woven Cotton Tapes for Electrical Purposes (D 335 - 48 T),[4]

Tentative Method of Test for Stretch of Hosiery (D 1058 - 49 T),[4]

Tentative Methods of Testing Single Kraft Yarn (D 1057 - 49 T),[4] and

Tentative Specifications for Mechanical Sheet Felt (D 1114 - 50 T):[2]

The committee recommends that the above four tentative specifications and methods be approved for reference to letter ballot of the Society for adoption as standard without revision.

REVISION OF STANDARDS, IMMEDIATE ADOPTION

Committee D-13 recommends that the following two standards be revised as indicated and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that these recommendations may be referred to letter ballot of the Society:

Definitions of Terms Relating to Textile Materials (D 123 – 50):[2]

In Table II, revise Part C on Vegetable Fibers, seed and fruit hair fibers, to read as shown in the accompanying table.

Several editorial changes in Section D of Table II will also be incorporated in Standard D 123.

TABLE II.—NATURAL FIBERS

C. VEGETABLE FIBERS—SEED AND FRUIT HAIR FIBERS.

Commercial and Botanical Names[a]		Staple Length, in.	Geographical Sources
Akund	*Calotropis gigantes*		Asia
	Gossypium, various sp		
COTTON			
1. Asiatic type:			
a. Chinese	*Gossypium arboreum*	⅜ to 1	China
b. Indian	*Gossypium arboreum and Gossypium herbaceum*	⅜ to 1	India
c. Levantine	*Gossypium herbaceum*	½ to 1	Asia
2. Upland type	*Gossypium hirsutum*	¾ to 1 7/16	All Continents
3. Long fiber type:			
a. Sea Island	*Gossypium barbadense*	1½ to 2½	West Indies
b. Egyptian	*Gossypium barbadense*	1⅛ to 1⅝	Egypt, Sudan, United States, Peru
4. Peruvian type:			
a. Semi-rough	*Gossypium barbadense*	1⅛ to 1⅜	
Tanguis			Peru
Ishan			Africa
b. Rough	*Gossypium barbadense*	1¼	Peru
5. American Tree Cotton	*Gossypium hirsutum var. Marie Galante*		
Marie Galante		1¼	British West Indies
Haiti (Bourbon)		1¼	Haiti
Moco		1¼	Brazil
KAPOK	*Ceiba pentandra*	¾ to 1¼	Tropics
MILKWEED FLOSS	*Asclepias, various sp*	¾ to 1¼	North America
Ozone fiber	*Asclepias sp. incarnata*		United States
Pochote	*Ceiba aesculifolia*		Mexico
Samchu	*Chorisia species*		South America

[a] Preferred or most common names are in capital letters; botanical names are in italics.

Standard Methods of Testing Felt (D 461 – 50):[2]

On the recommendation of Subcommittee A-10, the following revision in this standard is recommended for immediate adoption as this change has already been made in Government specifications:

Section S6 (d).—Change the time of immersion from "60 min" to "20 min."

WITHDRAWAL OF STANDARD AND TENTATIVE

Tentative Specifications for Bleached Cotton Broadcloth (D 504 – 41 T), and Standard Specifications for Terry (Turkish) Toweling (D 505 – 40):

The committee recommends the withdrawal of the standard and tentative listed above.

EDITORIAL CHANGES

Tentative Specifications and Methods for Asbestos Yarns (D 299 – 50 T),[2] **Standard Specifications and Methods of Test for Asbestos Roving for Electrical Purposes (D 375 – 49),**[4] **and Standard Specifications for Woven Asbestos Cloth (D 677 – 50):**[2]

On the recommendation of Subcommittee A-4, the following editorial changes in the above specifications are to be made: In the fourth column of Table I of Methods D 299 and D 375 and of the Table in Section 9 of Specifications D 677, opposite 7-cut change "581 to 819" to "595 to 805," and opposite 50-cut change "4275 to 5750" to "4250 to 5750."

REPORT OF COMMITTEE D-14

ON

ADHESIVES*

Committee D-14 on Adhesives held two meetings during the year. A two-day meeting was held in Boston, Mass., on October 19 and 20, 1950, at which a talk, "Investigations of Engineering Adhesives by Means of Ultrasonics," was given by A. G. H. Dietz of Massachusetts Institute of Technology. On April 23 and 24, 1951, a meeting was held in Washington, D. C., at the National Bureau of Standards. J. J. Bikerman of Merck & Co., was the guest speaker and discussed some aspects of adhesion and tack.

At the present time, Committee D-14 consists of 82 members, of whom 33 are classified as producers, 35 as consumers, and 14 as general interest members.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1950 Annual Meeting, Committee D-14 presented to the Society through the Administrative Committee on Standards the following recommendations:

Tentative Recommended Practice for:

Determining Strength Development of Adhesive Bonds (D 1144 – 51 T), and

Determining the Effect of Moisture and Temperature on Adhesive Bonds (D 1151 - 51 T).

Tentative Method of:

Determining the Blocking Point of Potentially Adhesive Layers (D 1146 – 51 T).

The tentative Recommended Practice D 1144 – 51 T was accepted by the Standards Committee on January 19, 1951, Tentative Recommended Practice D 1151 – 51 T on May 26, 1951, and Tentative Method D 1146 – 51 T was accepted on March 2, 1951. The new tentatives appear in the 1951 Supplement to Book of ASTM Standards, Part 4.

There is presently under consideration by the Administrative Committee on Standards the proposed Tentative Test for Strength Properties of Adhesives on Flexural Loading, and the proposed Tentative Method of Test for Resistance of Adhesives for Wood to Cyclic Accelerated Service Conditions.[1]

NEW TENTATIVE

Committee D-14 recommends the publication of a new Tentative Method of Determining the Effect of Bacterial Contamination on the Permanence of Adhesive Preparations and Adhesive Bonds, as appended hereto.[2]

ADOPTION OF TENTATIVES AS STANDARDS

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Methods of Test for:

Resistance of Adhesive Bonds to Chemical Reagents (D 896 – 50 T),[3]

Applied Weight per Unit Area of Dried Adhesive Solids (D 898 – 47 T),[4]

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* Presented at the Fifty-fourth Annual Meeting of the Society, June 18–22, 1951.

[1] See Editorial Note, p. 406.

[2] The new tentative was accepted by the Society and appears in the 1951 Supplement to Book of ASTM Standards, Part 4.

[3] 1950 Supplement to 1949 Book of ASTM Standards, Part 4.

[4] 1949 Book of ASTM Standards, Part 4.

Applied Weight per Unit Area of Liquid Adhesive (D 899 - 47 T),[4] and

Cleavage Strength of Metal-To-Metal Adhesives (D 1062 - 49 T).[4]

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends that the following tentatives be continued in their present status without revision, pending further evaluation work and revision:

Recommended Practice for Determining the Effect of Artificial (Carbon-Arc Type) and Natural Light on the Permanence of Adhesives (D 904 - 46 T).

Definition of Terms Relating to Adhesives (D 907 - 49 T).

Method of Test for Strength Properties of Metal-to-Metal Adhesives in Shear by Tension Loading (D 1002 -49 T).

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[5]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Strength Properties (C. B. Hemming, chairman).—A new, simple type of tension test is the basis for a new method being considered by the Section on Tensile Strength.

The Section on Shear Strength has distributed, for examination by Committee D-14, a proposed method on rigid type plastic to plastic bonding with round-robin data bearing on this method.

A revision of Tentative Method of Test for Impact Strength of Adhesives (D 950 - 47 T) by the Section on Impact Strength has been recommended for submission to letter ballot of Committee D-14.

A method of test for strength properties of adhesives on flexural loading, prepared by the Section of Flexural Strength, has been recommended by letter ballot of Committee D-14 and is now before the Administrative Committee on Standards.[1]

The Sections on Fatigue and Peel Tests have been active, as has been the recently reorganized Section on Creep and Flow.

Subcommittee II on Analytical Tests (L. Repsher, chairman).—The subcommittee has concluded extensive round-robin tests on various methods of determining the nonvolatile content of urea resin adhesives. One of these methods has been chosen for letter ballot of the subcommittee.

Subcommittee III on Permanence (R. F. Blomquist, chairman).—Two methods, presented by the Section on the Effect of Moisture and Temperature, were recommended by letter ballot of Committee D-14. One of these methods, Method D 1151 - 51 T, has been accepted and the other is now before the Administrative Committee on Standards, as noted earlier in the report.

Method D 896 - 50 T presented by the Section on Effect of Chemical Reagents has been recommended by Committee D-14 for adoption as standard by the Society.

The Section on Biological Factors has presented a method "Determining the Effect of Bacterial Contamination on the Permanence of Adhesive Preparations and Adhesive Bonds" which is referred to earlier in this report.

Further consideration is being given to revision of Method D 904 - 46 T by the Section on Effect of Light on Permanence.

The Section on Correlation of Short Term and Long Term Tests is considering means of utilization of the ASTM Test Sites being made available.

Subcommittee IV on Working Properties (D. L. Swayze, chairman).—The Section on Tack and Blocking has presented a Method on Blocking (D 1146 - 51 T) which has been recommended by letter ballot of Committee D-14 and accepted by the Administrative Committee on Standards. Several members who now have tack testing instruments have met and outlined round-robin tests of tackiness.

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[5] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

A Recommended Practice for Determining the Rate of Strength Development of Adhesive Bonds (D 1144 - 51 T), presented by the Section on Rate of Strength Development, has been accepted by the Administrative Committee on Standards, as noted earlier in the report.

Work is continuing in the Sections on Slippage and Flow, Storage and Working Life. The Section on Penetration is being reviewed as to whether or not it should remain inactive. The Section on Gap Filling Properties has been inactivated.

Subcommittee V on Specifications (F. H. Bair, chairman).—The Section on General Utility Adhesives has discussed hand label and mending adhesives. Further round-robin tests will be made on adhesives for acoustical materials in accordance with methods agreed upon by the Section to collect more data to support proposed specifications.

The Section on Brake Shoe Adhesive (originally Section F) has been discontinued pending work in ASTM Committee D-11. A new Section F on Wood-to-Wood Adhesives has been organized.

Subcommittee VI on Nomenclature (F. Moser, chairman).—A definition of permanence was approved by Committee D-14. Work is under way on definitions for: plasticizer, plasticize, consistency, emulsion, dispersion, thixotrophy, syneresis, diluent, polar, doctor bar or blade, doctor rolls, pick-up rolls, warp, creep, solution, yield value, plasticity, wetting agent, primer, and viscosity.

Subcommittee VII on Research (R. C. Platow, chairman).—The Research Subcommittee has discussed pure shear, the program on the investigation of wood adhesives at the University of Michigan, what constitutes adhesion, and the testing of adhesives at very high temperatures.

Subcommittee VIII on Electrical Properties (H. A. Perry, chairman).—A proposed general method of test for the electrical properties of adhesives is being circulated for comment.

This report has been submitted to letter ballot vote of the committee, which consists of 82 voting members; 52 members have returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

FRANK W. REINHART,
Chairman.

L. M. PERRY,
Secretary.

EDITORIAL NOTE

On May 26, 1951, the Administrative Committee on Standards accepted the recommendation of Committee D-14 that the Recommended Practice for Determining the Effect of Moisture and Temperature on Adhesive Bonds be published as tentative. The tentative Recommended Practice appears in the 1951 Supplement to Book of ASTM Standards, Part 4, bearing the designation D 1151 - 51 T.

Subsequent to the Annual Meeting, Committee D-14 presented to the Society through the Administrative Committee on Standards the following recommendations:

Tentative Methods of:

Test for Resistance of Adhesives for Wood to Cyclic Accelerated Service Conditions (D 1183 - 51 T), and
Test for Strength of Adhesives on Flexural Loading (D 1184 - 51 T).

These recommendations were accepted by the Standards Committee on July 10, 1951, and the methods appear in the 1951 Supplement to Book of ASTM Standards, Part 4.

REPORT OF COMMITTEE D-15

ON

ENGINE ANTIFREEZES*

Committee D-15 on Engine Antifreezes held two meetings during the year: the first in New York, N. Y., on October 20, 1950, and the second in Washington, D. C., on March 30, 1951. The Advisory Committee and subcommittees have held meetings concurrently. There have been eight resignations and nine additions to the list of committee members during the year. At present the committee is composed of 40 active members, of whom 18 are classified as producers, 10 as consumers, and 12 as general interest members.

The following changes in subcommittee chairmanships occurred during the year: E. G. Travis vice R. J. Harker as chairman of Subcommittee IV on Chemical Properties; E. F. Harford vice E. H. Keller, chairman on Subcommittee VI on Simulated and Actual Service Testing; C. H. Sweatt vice J. C. Kratzer as chairman of Subcommittee VI's Study Group on Test Methods. R. E. Mallonee, after having been elected to the Advisory Committee to replace J. C. Kratzer, was elected as vice-chairman of Committee D-15 to complete the term of D. B. Brooks, resigned; J. M. Clark was elected to the Advisory Committee to fill the vacancy left by D. B. Brooks; C. F. Graham was elected secretary of Committee D-15 to replace R. J. Harker.

NEW TENTATIVES

Committee D-15 recommends that the following two methods be accepted for publication as tentative, as appended hereto:[1]

Method of Test for:

Freezing Point of Aqueous Engine Antifreeze Solutions.

Method for:

Sampling and Preparing Aqueous Solutions of Engine Antifreeze.

REVISION OF TENTATIVE

Tentative Method of Test for Boiling Point of Engine Antifreezes (**D 1120 – 50 T**).[2]—The committee recommends revision in this method as follows:

Section 1.—Revise this section on "Scope" to read as follows by the addition of the italicized words:

1. *Scope.*—This method of test is designed for the determination of the equilibrium boiling point of engine antifreezes that are miscible with water. The equilibrium boiling point indicates the temperature at which the sample will start to boil in a cooling system under *equilibrium conditions at* atmospheric pressure.

Omit the present Note in Section 1.

Section 4.—Revise to read as follows:

4. (*a*) *Sample.*—To obtain a sample of unused concentrated antifreeze for boiling point determination, secure the sample only from the major phase after the antifreeze has stood undisturbed in a closed container for 3 hr.

(*b*) To prepare dilute solutions of either one-phase or two-phase antifreezes, thoroughly mix the sample, pipet the required volume of antifreeze into a calibrated volumetric flask, and add distilled water to bring the mixed contents of the flask to the proper volume, while maintaining the calibration temperature. Allow to stand for 3 hr., then obtain the sample only from the major phase if two phases are present.

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* Presented at the Fifty-fourth Annual Meeting of the Society, June 18–22, 1951.

[1] The new tentatives were accepted by the Society and appear in the 1951 Supplement to Book of ASTM Standards, Part 5.

[2] 1950 Supplement to 1949 Book of ASTM Standards, Part 5.

Section 8.—Revise Section 8 on "Precision" to read as follows:

8. (*a*) *Reproducibility.*—For diluted samples boiling below 212 F, results should not differ from each other by more than 2.5 F and, for samples boiling above 212 F, results should not differ from each other by more than 4.5 F.

(*b*) For unused concentrated samples boiling below 212 F, results should not differ from each other by more than 2.5 F, and, for samples boiling above 212 F, results should not differ by more than 10 F.

Tentatives Continued Without Revision

The committee recommends that the following tentatives be retained as tentative without revision:

Specifications for:

Hydrometer-Thermometer Field Tester for Engine Antifreezes (D 1124 - 50 T).

Methods of Test for:

Ash Content of Concentrated Engine Antifreezes (D 1119 - 50 T),

Reserve Alkalinity of Concentrated Engine Antifreezes (D 1121 - 50 T),

Specific Gravity of Concentrated Engine Antifreezes by the Hydrometer (D 1122 - 50 T), and

Water in Concentrated Engine Antifreezes by the Iodine Reagent Method (D 1123 - 50 T).

The recommendations appearing in this report have been submitted for letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

Activities of Subcommittees

Subcommittee I on Freezing Point Determination (R. E. Mallonee, chairman) prepared the proposed Tentative Method of Test for Freezing Point on Aqueous Antifreeze Solutions. Results of the final cooperative tests made in the development of this method are shown in Table I.

TABLE I.—FREEZING POINTS OF TEST SOLUTIONS IN DEGREES FAHRENHEIT, USING PROPOSED FREEZING POINT METHOD.

		Sample C-1-1		Sample C-2-2		Sample C-5-2	
		Solution No. 1	Solution No. 2	Solution No. 1	Solution No. 2	Solution No. 1	Solution No. 2
E. I. duPont deNemours Co.		9.3	−56.2	−0.8	−35.9	7.3	−43.6
Kimble Glass Co.		8.6	−55.7	−1.8	−36.8	6.3	−43.8
National Carbon Div., UCC		8.8	−55.3	−1.4	−37.6[a]	6.5	−44.3
The Texas Co.		8.9	−56.1	−1.6	−36.3	6.7	−44.2
U. S. Industrial Chemicals, Inc.		8.7	...	−1.7	−35.0[a]	6.4	...
Mean Value for Freezing Point		8.9	−55.8	−1.5	−36.3	6.6	−44.0
Maximum Deviations from Mean	Plus	0.4	0.4	0.3	1.3	0.7	0.3
	Minus	0.3	0.5	0.7	1.3	0.3	0.4
	Range	0.7	0.9	1.0	2.6	1.0	0.7

[a] Dropping either the NCD or USI value for solution No. 2 of Sample C-2-2 brings the range below 2.0 F.

Subcommittee III on Physical Properties (R. E. Vogel, chairman) established the precision limits for the Tentative Method of Test for Specific Gravity of Concentrated Antifreezes by the Hydrometer (D 1122 - 50 T) with a reproducibility of ±0.0012, which were included in the method as published last year. The results of the cooperative tests are shown in Table II. Several revisions were approved and are recommended for the boiling point procedure (D 1120 - 50 T) for purposes of clarity.

Subcommittee IV on Chemical Properties (E. G. Travis, chairman) is studying the revisions necessary in the existing Method for Determination of pH of Aqueous Solutions with a Glass Electrode (E 70 - 46 T) to make it applicable for the determination of pH of aqueous antifreeze solutions. The subcommittee is also preparing methods to test for classification of engine antifreezes as to type. At present, the five classifications decided upon include: water-soluble low-boiling types; water-soluble high-boiling types; salt-base types; petroleum or water-insoluble types; and others.

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[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee V on Effects of Antifreeze on Rubber Hose (T. E. Bieterman, chairman) held its first meeting on March 29, 1951. A testing program was outlined whereby six cooperating laboratories are to run the SAE-ASTM immersion test procedure along with their own type of rubber test, using three grades of rubber and standard coolant furnished by the committee.

TABLE II.—DETERMINATION OF SPECIFIC GRAVITY OF CONCENTRATED ANTIFREEZE BY MEANS OF THE HYDROMETER AT 60/60 F.

	Sample C-1-2	Sample C-2-2[a]	Sample D-3-2	Sample E-4-2
Carbide and Carbon Chemicals	0.8010	1.1260	0.8157	0.8190
E. I. duPont de Nemours Co.	0.8010	1.1256	0.8164	0.8200
Enjay Co.	0.8000	1.1260	0.8160	0.8195
Gulf Oil Co.	0.8008	1.1260	0.8160	0.8193
Kimble Glass Co.	0.8013	1.1259	0.8169	0.8206
Montgomery Ward	0.7998	1.1260	0.8153	0.8193
Packard Motor Co.	0.8010	1.1241	0.8173	0.8205
Shell Oil Co.	0.8000	1.1250	0.8158	0.8190
Socony-Vacuum Co.	0.8010	1.1252	0.8168	0.8193
The Texas Co.	...	...	0.8156	0.8182
U. S. Industrial Chemicals, Inc.	0.8008	1.1243	0.8164	0.8199
U. S. Naval Engineering Experiment Station	0.8012	...	0.8167	0.8203
Average	0.8008	1.1253	0.8162	0.8195
High	0.8013	1.1260	0.8173	0.8206
Low	0.7998	1.1241	0.8153	0.8182
Range	0.0015	0.0019	0.0020	0.0024

[a] Oil layer separated and specific gravity measured on lower layer.

Subcommittee VI on Simulated and Actual Servicing Testing (E. F. Harford, chairman). The Study Group on Simulated and Actual Servicing Testing (C. H. Sweatt, chairman) has prepared a report summarizing the collaborative work from the glassware bench test and the modified Bureau of Standards circulating test for the antifreeze corrosion study. It has been concluded that because of the limitations of the glassware test it should be considered only as a screening tool at this time, but that it should also be developed further. With reference to the circulating test, the mass of data is so great that it will take some time to complete the study. Further testing work will await the completion of this study. The Study Group on Preparation of Antifreeze and Cooling System Recommendations (Gus Kaufman, chairman) has prepared an article on the Selection and Use of Engine Antifreeze, which has been approved by the committee and which will be submitted to the Society shortly for publication.

Subcommittee VII on Specifications (J. M. Clark, chairman) is preparing the general outline form for an antifreeze performance specification using the tests as developed in the other subcommittees. However, this subcommittee does not currently have available sufficient data to prepare actual specification limits for engine antifreezes. It is depending upon the projects under investigation by the other subcommittees to provide the necessary information for the specification limits.

Study Group on Sampling (R. E. Mallonee, chairman) prepared the proposed Tentative Method for Sampling and Preparing Aqueous Solutions of Engine Antifreezes.

This report has been submitted to letter ballot to the committee, which consists of 40 voting members; 32 members have returned their ballots, of whom 30 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

H. R. WOLF,
Chairman.

C. F. GRAHAM,
Secretary.

REPORT OF COMMITTEE D-16

ON

INDUSTRIAL AROMATIC HYDROCARBONS*

Meetings of the Advisory and General Membership of Committee D-16 on Industrial Aromatic Hydrocarbons were held on June 27, 1950, at Atlantic City, N. J., during the 1950 Annual Meeting of the Society, and on March 6, 1951, at Cincinnati, Ohio, during the ASTM Spring Group Committee Meetings.

In view of the completion of the original objective of this committee, considerable attention has been given to the matter of increasing the scope of activities and of defining the limits of such expansion. The basic idea in the expansion is the inclusion of such materials as phenol and its homologues, pyridine and its homologues, naphthalene, and other materials which are logically considered after the industrial aromatic hydrocarbons. Committee approval was expressed by a preliminary vote, but no formal recommendation has as yet been made to the Board of Directors. This proposed expansion of activities would involve an extension in personnel, and a membership committee has been appointed to develop the matter. Initial activity will deal with methods of test, and assistance of qualified personnel will be welcomed.

Section F of Subcommittee 5 was established, under the chairmanship of A. W. Beshgetoor, to consider the problem of olefin contamination of benzene produced by new processes.

ADOPTION OF TENTATIVE AS STANDARD

Committee D-16 recommends that the Tentative Methods of Test for Specific Gravity of Industrial Aromatic Hydrocarbons (D 891 - 46 T)[1] be approved for reference to letter ballot of the Society for adoption as standard, with the addition of the following note to Section 1 (*b*): "NOTE 3.—In referee problems, the pycnometer method (Method C) shall be used."

The recommendation appearing in this report has been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

This report has been submitted to letter ballot of the committee, which consists of 38 voting members; 25 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee.

D. F. GOULD,
Chairman.

R. B. GREENE,
Secretary.

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[*] Presented at the Fifty-fourth Annual Meeting of the Society, June 18-22, 1951.

[1] 1949 Book of ASTM Standards, Part 5.

[2] The letter ballot vote on this recommedation was favorable; the results of the vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE D-17

ON

NAVAL STORES*

Committee D-17 on Naval Stores has held two meetings during the past year: one in connection with the Annual Meeting of the Society on June 29, 1950, at Atlantic City, N. J., and the other on March 8, 1951, during the Spring Committee Meeting week at Cincinnati, Ohio.

At both meetings, a thorough discussion was held of the work of each subcommittee, in lieu of separate subcommittee meetings. Although this procedure causes the general meeting to be of longer duration, the work of the subcommittees and the entire committee program is more thoroughly understood and reviewed by all members in attendance at the meeting.

On the strength of favorable discussion at the June, 1950, meeting of the suggestion to prepare ASTM specifications for some other naval stores products, in addition to the standard specifications for turpentine, the Advisory Committee has approved the formation of a new Subcommittee on Specifications. S. R. Snider will assume the chairmanship of the new subcommittee. The preparation of ASTM specifications on naval stores must, of necessity, take into consideration the fact that many, if not most, types of naval stores products other than turpentine and rosin are manufactured in different qualities or grades to fit the peculiar needs of the individual consumer. This leads to special producer-consumer relations not fully dependent on any standardized general specifications.

On behalf of Committee D-17, W. A. Kirklin, in a report to Committee E-12 on Appearance, reviewed the various aspects of color testing as it relates to naval stores products. It was recommended that Committee E-12 give consideration to the possibility of describing colors and color grades of industrial materials in general by reference to the I.C.I. system of color nomenclature and measurement, based on fundamental units of color values.

The committee lost two consumer members during the year, one by death, W. C. Bainbridge, of H. Kohnstamm and Co., and one by resignation. Two new members were added to the committee, both of whom represent the tall oil producing interests. The over-all membership of the committee remains unchanged.

TENTATIVE REVISION OF STANDARD

In view of the major changes being proposed in a number of the standard definitions for terms relating to naval stores, Committee D-17 recommends the publication as tentative of the revised and new definitions as appended hereto.[1] These definitions are in effect a tentative revision of the Standard Definitions of Terms Relating to Naval Stores and Related Products (D 804 – 48).[2] These definitions have had the benefit of thorough discussion at the last general meeting of the committee and have been approved for such acceptance by letter ballot of the entire committee.

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* Presented at the Fifty-fourth Annual Meeting of the Society, June 18–22, 1951.

[1] The new tentative was accepted by the Society and appears in the 1951 Supplement to Book of ASTM Standards, Part 4.

[2] 1949 Book of ASTM Standards, Part 4.

ADOPTION OF TENTATIVES AS STANDARD

Several of the subcommittees have been actively studying the various test methods which have been tentative for a year or more. The committee recommends at this time that the following three tentative methods be referred to letter ballot of the Society for adoption as standard without revision:

Tentative Methods of Test for:
Tall Oil (D 803 - 49 T),[2]
Ash in Rosin (D 1063 - 49 T),[2] and
Iron in Rosin (D 1064 - 49 T).[2]

Recommendation is also made that the following four tentatives be referred to letter ballot of the Society for adoption as standard, with revisions as indicated:

Tentative Method of Test for Acid Number of Rosin (D 465 - 49 T):[2]

Section 2.—Insert the following new section on sample preparation, and renumber subsequent sections accordingly:

> 2. *Preparation of Sample.*—The sample taken for analysis shall consist of small pieces of rosin chipped from a freshly exposed part of a lump or lumps, and thereafter crushed to facilitate weighing and solution. The sample shall be so prepared the same day on which the test is begun, in order to avoid changes in properties due to surface oxidation, which is very pronounced on ground rosin having a large surface area exposed to the air.

Section 3 (b).—Change to read as follows:

> (*b*) *Standard Alkali Solution (0.5 N).*—Dissolve either 22 g of NaOH or 33 g of KOH (pellets or sticks) in water or in alcohol conforming to Paragraph (*a*), and dilute to 1 liter with the same solvent. Standardize to ±0.001 *N*.

Section 4 (b).—Change "NaOH" to "standard alkali," and add at the end of the sentence: "to the first permanent faint pink color."

Section 6 (a).—Delete the footnote reference 3 and the footnote, and add the following: "Use either standard or alkali-resistant electrodes for this test."

Section 9 (c).—Change to read as follows:

> (*c*) Titrate with the standard alkali solution. For normal rosin and with an alkali strength of not more than 0.52 *N*, 20 ml may be added initially to bring the pH of the test solution up to about 10.0. Record the pH and buret readings. Then add alkali in 1.0 ml portions until the change in pH equals or exceeds 0.3 pH units per ml. From this point reduce the additions of alkali to 0.1 ml until the end point has been passed, as indicated by a significant decrease in pH change per 0.1 ml addition. Then add 1.0-ml portions until a pH of 12 is recorded.

Section 9 (d).—Change "0.2 ml." to "0.1 ml" and "NaOH" to "alkali." Insert "0.1" before "ml" in the next to last sentence.

Tentative Method of Test for Saponification Number of Rosin (D 464 - 49 T).[2]—This method has also been revised to incorporate a section on sample preparation and a potentiometric titration procedure suitable for use with dark-colored rosin:

Section 2.—Add a new section on sample preparation, to read the same as the proposed revision of Section 2 in Method D 465, and renumber subsequent sections accordingly.

Section 7 (a).—Delete the footnote reference 4 and the footnote, and add the following sentence: "Use either standard or alkali-resistant electrodes for this test".

Section 10 (d).—Change to read as follows:

> (*d*) Titrate with the standard sulfuric acid. If the normality factor is not above 0.52 *N*, 20 ml may be initially added, which should not reduce the pH below 13. Record the pH and buret readings. (A slight increase in pH in the early stage of the titration results from liberation of hydroxyl ions due to hydrolysis of the

soap. The first significant break in the titration curve occurs shortly after this observation.) Now add acid in 1.0-ml portions so long as the change in pH does not exceed 0.3 pH units. Then reduce the additions to 0.1-ml portions until the end point has been passed, as indicated by a significant decrease in pH change per 0.1-ml addition. After this has been established, add acid in 1.0-ml portions until a pH of 8 is reached.

Section 10 (e).—Change "0.2" to "0.1" in the two places where it appears in the first sentence and substitute "0.1 ml" in place of the word "milliliter" in the second sentence.

Tentative Method of Test for Water in Liquid Naval Stores (D 890 - 50 T).[3]—The revised method embodying a change in the Karl Fischer reagent to a two—solution reagent has been found to have distinct advantages, and is ready for advancement to standard with the following editorial changes:

Section 1 on Scope.—Add the following note at the end of this section:

NOTE.—The Karl Fischer reagent prescribed in this test is prepared in two solutions, in which form they are more stable and less affected by atmospheric moisture. However, the single solution reagent described in Tentative Method of Test for Water in Concentrated Engine Antifreezes by the Iodine Reagent Method (ASTM Designation: D 1123) may be used if it is more readily available. The solvents and proportions specified in Method D 890, should be employed if the single solution reagent is used.

Section 3 (a).—Add the following note at the end of this paragraph:

NOTE.—Electrometric titration may be used in lieu of visual observation of the end point.

Tentative Method of Test For Unsaponifiable Matter in Rosin (D 1065 - 49 T).[2]—The following changes are proposed for incorporation in this method:

Section 2.—Add a new Section 2 on Preparation of Sample, to read as included in the proposed revisions of Method D 465, and renumber subsequent sections.

Section 2.—Add the following as a new Paragraph (*e*): "(*e*) *Spot Plate*.—A Coors white porcelain spot plate."

Section 4 (e).—Change to read as follows: "*Thymol Blue Solution*.—Dissolve 0.1 g thymol blue in 100 ml of methanol."

Section 4 (g).—Delete.

Section 5 (a).—Add the words "without boiling" after "evaporate" in the last sentence.

Section 5 (d).—In second sentence, change "100 mm of mercury or less" to read "100 ± 10 mm of mercury." Add the following note:

NOTE.—When a vacuum oven is not available, evaporate the ether extract in a weighed 200-ml round-bottom flask, on a steam bath. Complete the volatilization by placing the flask in a water bath at 70 C, connect to a vacuum pump or aspirator, and aspirate for 1 hr at 100 ±10 mm of mercury. Wipe the flask, cool in desiccator, and weigh as prescribed."

Section 5 (e).—Change to read as follows:

(*e*) Dissolve contents of flask in 40 ml of neutral ethyl alcohol, add 1 ml of thymol blue indicator solution, and titrate with 0.1 *N* alcoholic KOH solution. When the solution is too colored to accurately detect the end point internally, titrate until a faint color change is noted. Then withdraw approximately 0.5 ml of the solution to the porcelain spot plate, and to the withdrawn portion add 1 drop of the thymol blue indicator solution. Continue titrating with 0.1-ml portions of alkali and testing until a deep blue color, which persists for at least 15 sec, indicates that the end point has been reached.

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting of the Society.[4]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Softening Point of Rosin (J. M. Schantz, chairman).—The representative of this subcommittee, serving as chairman of the Task Group on Softening Point of Committee E-1, reports further progress in the group's efforts to effect a combination into one standard of the two present ASTM Ring-and-Ball Softening Point Methods, D 36 - 26 and E 28 - 42 T. A proposed revised tentative method has been prepared which includes a description of the stirring device to be used and a technique for preparing the ring sample of resins (excepting rosin) and other materials which cannot be melted, by compressing the powdered material into the ring.

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[3] 1950 Supplement to Book of ASTM Standards, Part 4.

[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee III on Volatile Oils in Rosin (S. R. Snider, chairman).—A test was made to determine whether there is any tendency for a variation in the volatile oil content of the rosin in different parts of a commercial drum, (about 520 lb, net, of rosin) due to "layering" as the rosin cools and solidifies. For this test a special small diameter, standard height rosin drum was filled with an incompletely processed or partially distilled gum rosin, containing about eight times the volatile oil (turpentine) content usually found in gum rosin. Samples for the test were taken from the top, middle, and bottom of the drum after it had cooled and was knocked down to get to the rosin. No variation was found in the oil content of the three samples.

Subcommittee IV on Chemical Analysis of Rosin (W. D. Pohle, Chairman).—On the basis of additional study by a number of collaborating laboratories of the potentiometric methods for titrating acid number of rosin, and for back-titrating the excess alkali in the saponification number test, on both light and dark rosin, the subcommittee has again recommended adoption as standard of the tentative methods now published for these determinations. The recommendation incorporates the proposed revisions published in the 1950 annual report, along with a rewording of the directions for carrying out the potentiometric titrations. The subcommittee further recommends adoption as standard of the Tentative Method of Test for Unsaponifiable Matter in Rosin (D 1065 - 49 T) with changes as indicated previously in this report. Further study is planned for the proposed method for unsaponifiable matter in rosin using separatory funnels in place of the continuous extraction apparatus. This method has not given reliable results heretofore on wood rosins.

Subcommittee V on Tall Oil (A. Pollak, chairman).—The present tentative methods have been given further study by members of the subcommittee and other cooperating laboratories. It is now recommended that the present methods be adopted as standard. Tall oils of very low rosin acid content (up to 10 per cent) do not behave exactly like normal tall oils that contain more nearly equal parts of rosin acid and fatty acid, when subjected to the modified Wolfe esterification procedure for determining the rosin acid number. A number of laboratories dealing with tall oil are cooperating in an endeavor to devise an alternate or substitute method for rosin acid number of highly refined tall oil. When a promising method has thus been developed, further collaborative work will be done on known mixtures of rosin acids and fatty acids. Such alternate rosin acids method will be proposed for eventual addition to the present methods now being recommended for adoption as standard.

Subcommittee VII on Terpene Hydrocarbons and Pine Oil (R. E. Price, chairman).—The two-solution modification of the Karl Fischer method for Determination of Water in Liquid Naval Stores (D 890 - 50 T) published as a tentative last year has been given further collaborative study by five laboratories, using samples of steam-distilled wood turpentine, gum spirits of turpentine, and pine oil. Excellent agreement was obtained on the gum turpentine and pine oil samples. In the case of the steam-distilled wood turpentine, which was a freshly made turpentine almost free from moisture, the agreement between several laboratories was not good. Subsequent reruns by two analysts of the analysis on the same samples after standing several months, showed a marked increase in the moisture content (or peroxides). This indicates that the moisture determination by this method must be made on a sample held in a completely filled container shortly after it is taken, and that a repeat analysis made on an oxidizable material such as turpentine cannot be depended upon if it has been allowed to stand exposed to air in a partially filled container. The resulting oxidation generates moisture or peroxides which react as moisture with the iodine reagent.

Subcommittee IX on Definitions (J. L. Boyer, chairman).—The subcommittee has reviewed the definitions for naval stores and related products. The promulgation of a new Federal standard of nomenclature and identity for "tall oil rosin," made from tall oil by a high-vacuum distillation process, and the use of tall oil as a source of rosin acids in place of rosin for some uses, has made it necessary to revise some of the present definitions. Several new definitions are also being proposed as mentioned earlier in this report.

This report has been submitted to letter ballot of the committee, which consists of 39 voting members; 24 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

V. E. GROTLISCH,
Chairman.

W. A. KIRKLIN,
Secretary.

REPORT OF COMMITTEE D-18

ON

SOILS FOR ENGINEERING PURPOSES*

Committee D-18 on Soils for Engineering Purposes held two meetings during the year. The first meeting was held during the Annual Meeting at Atlantic City, N. J., on June 30, 1950. At this meeting the following officers were elected for the ensuing term of two years:

Chairman, E. J. Kilcawley,
First Vice-Chairman, W. S. Housel,
Second Vice-Chairman, F. J. Converse,
Secretary, W. G. Holtz, and
Assistant-Secretary, E. A. Abdun-Nur.

The committee held a second meeting in Cincinnati, Ohio, on March 8, 1951. After the business meeting, the following papers were presented:

"Identification of Clay Minerals by Staining Test," by R. C. Mielenz and M. E. King, presented by Mr. Mielenz,[1]

"An Analysis of the Effect of Particle Interlocking on the Strength of Cohesionless Soil," by B. K. Hough,[2] presented by the author, and

"De-aired, Extruded Soil Specimens for Research and for Evaluation of Test Procedures," by L. Hudson Matlock, Jr., Carl Fenske, and Raymond F. Dawson,[3] presented by Mr. Dawson.

A separate Executive Subcommittee meeting was held on January 8, 1951, in Washington, D. C.

During the year, six members were added to the committee; there were four separations. The total committee membership is now 141, which includes 101 voting memberships and 20 consulting memberships.

The committee suffered the loss, through death, on May 10, 1951, of Edward Allen Willis, a valued and active member. Mr. Willis, a senior highway engineer with the Bureau of Public Roads, contributed greatly through subcommittee participation and as a section chairman to the development of several standards on test methods for soils.

Two soils symposiums were held at the 1950 Annual Meeting. The first symposium, on Methods of Testing Soils under Triaxial Loading, occupied two sessions and consisted of ten papers. The second symposium, including five papers on the Identification and Classification of Soils, was presented in a third session. All of these papers and discussions are being published by the Society as special technical publications (STP Nos. 106, 113). In addition to the symposium in the third session, a paper entitled "Pilot Studies of Soil Density Measurement by Means of X-rays," by D. Bardan and R. K. Bernhardt, Rutgers University, was presented.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the Annual Meeting, Committee D-18 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Method of:

Test for Amount of Material Finer Than No. 200 Sieve for Soil (D 1140 – 50 T).

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* Presented at the Fifty-fourth Annual Meeting of the Society, June 18–22, 1951.

[1] See p. 1213.

[2] ASTM BULLETIN, No. 176, September, 1951, p. 55 (TP 193).

[3] Ibid, No. 177, October, 1951, p. 51 (TP 215).

Revision of Tentative Method of:

Test for Specific Gravity of Soils (D 854 - 45 T).

These recommendations were accepted by the Standards Committee on September 26, 1950, and the new and revised tentatives appear in the 1950 Supplement to Book of ASTM Standards, Part 3.

On December 14, 1950, the Administrative Committee on Standards accepted the recommendation of Committee D-18 that the Method of Test to Determine the Load Settlement Relationship for Individual Piles be published as tentative. The new tentative method appears in the 1950 Supplement to Book of ASTM Standards, Part 3, bearing the designation D 1143 - 50 T.

NEW TENTATIVES

Committee D-18 has under preparation the following proposed methods of test, which will be submitted to the Administrative Committee on Standards for acceptance as tentative subsequent to the Annual Meeting:

Tentative Methods of Test for:

Bearing Capacity for Soil for Static Load on Spread Footings,

Repetitive Static Load Tests for the Evaluation and Design of Airport and Highway Pavements, and

Nonrepetitive Static Load Tests for the Evaluation and Design of Airport and Highway Pavements.

TENTATIVE REVISIONS ADOPTED AS STANDARD

The committee recommends that the tentative revisions, pertaining to Sections 6(*b*) and 14, of the Standard Method of Mechanical Analysis of Soils (D 422 - 39)[1] be adopted as standard.

TENTATIVES CONTINUED WITHOUT REVISION

Committee D-18 recommends that the following tentatives be continued in their present status without revision:

Tentative Definitions of:

Terms and Symbols Relating to Soil Mechanics (D 653 - 42 T).

Tentative Methods of Test for:

Moisture-Density Relations of Soils (D 698 - 42 T), and

Shear Strength of Flexible Road Surfaces, Subgrades, and Fills by the Burggraf Shear Apparatus.

Tentative Method of Testing:

Soil-Bituminous Mixtures (D 915 - 47 T).

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee G-2 on Special Papers (R. F. Dawson, chairman) arranged a program of special papers for the Spring Committee Week in Cincinnati, Ohio. These papers are listed earlier in this report.

Subcommittee G-3 on Nomenclature and Definitions (C. R. Foster, chairman).—Approval, from the standpoint of soil mechanics nomenclature, was given to three proposed tentative load tests. The subcommittee plans to continue consideration of the proposed revisions to Tentative Definition Relating to Soils Mechanics (D 653 - 42 T) and, if possible, complete the work so that recommendations can made during the next year. This tentative has stood two years without revision.

Subcommittee G-5, Symposium (F. J. Converse, chairman).—Two symposiums on Triaxial Shear and Classification of Soils, respectively, were arranged for the 1950 Annual Meeting. Symposiums on

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[1] 1949 Book of ASTM Standards, Part 3.

[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Consolidation Testing of Soils and on Surface and Subsurface Reconnaissance are planned for presentation at the 1951 Annual Meeting.

Subcommittee R-1 on Surface and Subsurface Reconnaissance (L. E. Gregg, chairman) has directed its efforts toward the Symposium on Surface and Subsurface Reconnaissance to be presented at the 1951 Annual Meeting.[4] Preprinting of the papers and a panel discussion among the authors of papers have been stressed. In addition, equipment used in all phases of the reconnaissance will be displayed. Response by the authors has been outstanding, 15 papers having been obtained for discussion of the geological, geophysical, pedological, and air-photo methods of reconnaissance.

Subcommittee R-2 on Borings and Sampling for Soil Investigation (J. O. Osterberg, chairman). — Two new methods are being discussed by the subcommittee: one a Proposed Method on Borings and Sampling for Soil Investigation of Embankment Material, is now in its second revision; the other is a Proposed Method for Split-spoon and Thin Wall Tube Sampling of Soils. It is expected that the methods will be submitted to Committee D-18 for approval next year.

Subcommittee R-3 on Physical Characteristics of Soil (H. Allen, chairman).—During the year, Section A on Specific Gravity and Mechanical Analysis recommended for submission to letter ballot of Committee D-18 a proposed revision of Tentative Method of Test for Specific Gravity of Soils (D 845 - 45 T), a proposed Tentative Method of Test for Amount of Material Finer Than No. 200 Sieve for Soil, and incorporation into the standards of tentative revisions of Standard Method of Mechanical Analysis of Soils (D 442 - 39). Section A has also made progress in the study of possible revision of Method D 442.

Section B on Atterberg Limits has circulated a proposed revision of the liquid and plastic limit methods to Subcommittee R-3 and will proceed with a revision of these methods during the year.

Section C on Moisture-Density Tests has analyzed the returns of a questionnaire circulated on the use of the Tentative Method of Test for Moisture-Density Relations of Soils (D 698 - 42 T). Replies were received from 40 state highway departments, the District of Columbia, and 14 universities and commercial laboratories. The information received indicates that there is considerable variation from the procedure and that a revision is desirable. Such a revision will be undertaken during the coming year. The section will also undertake a study of methods of determining in-place density methods of soils.

Subcommittee R-5 on Structural Properties of Soil (D. M. Burmister, chairman).—The members of Section A were appointed as a special committee for the Symposium on Consolidation Testing to be held at the 1951 Annual Meeting.[5] The work of Section A on Consolidation Testing of Soils has consisted entirely of preparation and review of papers for presentation as part of the symposium.

Section B on Direct Shear has discussed a possible tentative for direct shear testing. To this end, a symposium of the subject is tentatively set for presentation at the 1952 Annual Meeting.

Section C on Triaxial Loading plans to study methods of conducting the triaxial shear test and the interpretation of the data obtained to date.

Subcommittee R-6 on Physico-chemical Properties of Soil (R. C. Mielenz, chairman).—Under the guidance of former chairman E. F. Preece, considerable progress has been made by the subcommittee in the formulation of a work program. Specific assignments for the development of procedures in connection with seven different methods of mineralogic and chemical analysis of soils have now been made to the subcommittee. Initial plans and procedures have been submitted to the chairman for: (*1*) complete chemical analysis of soils; (*2*) petrographic analysis of soils; and (*3*) preparation of hydrogen clays in quantity for laboratory use.

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[4] See p. 1260.

[5] See p. 1259.

The resignation of E. F. Preece from the chairmanship of the subcommittee and the appointment of R. C. Mielenz to the position is announced.

Subcommittee R-7 on Identification and Classification of Soils (G. W. McAlpin, chairman).—The chief activity of Subcommittee R-7 during the year was the preparation and presentation of a symposium on the identification and classification of soils, presented at the 1950 Annual Meeting. The purpose of the symposium was to bring before the Society a group of papers describing the most widely used procedures in current use for identifying and classifying soils for engineering purposes.

Subcommittee R-9 on Dynamic Properties of Soil (R. K. Bernhard, chairman).—The scope of the subcommittee is as follows: "Compilation, study, and correlation of data pertaining to dynamic properties of soils." A canvass has been made pertaining to research work referring to topics as mentioned in the scope outline. Characteristic dynamic properties of soils to be studied are elasticity, damping, stand and propagation waves, consolidation, settlement, densification, bedding values, etcetera, under dynamic loads, with or without static surcharges, and resonance phenomena of the vibratory systems. Methods to determine these dynamic properties are to be compared. Several research projects being carried out by various agencies are being followed, including: (*1*) a joint highway research project sponsored by the State Highway Department of New Jersey, the Public Roads Administration and Rutgers University; (*2*) a research project, sponsored by the Rensselaer Polytechnic Institute, on soil density determination by means of X-rays; (*3*) experiments with ultrasonic waves in soils referring to their propagation velocities; and (*4*) tests with pressure cells and the measurement of pile friction, a program started by the Association of American Railroads.

Subcommittee R-10 on Bearing Tests of Soil In Place (L. A. Palmer, chairman).—Section A on Soil Bearing Tests has prepared three tentative methods of test for plate loading tests for presentation to the committee membership for acceptance as tentative methods.

This report has been submitted to letter ballot of the committee which consists of 106 voting members; 69 members returned their ballots, of whom 66 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

E. J. KILCAWLEY,
Chairman.

W. G. HOLTZ,
Secretary.

REPORT OF COMMITTEE D-19

ON

INDUSTRIAL WATER*

Committee D-19 and its subcommittees held regular meetings June 28 to 30, 1950, at Atlantic City, N. J., and March 7 to 9, 1951, at Cincinnati, Ohio.

The committee is contributing a paper on flame photometry, by R. K. Scott, to the Symposium on Flame Photometry, to be presented at the 1951 Annual Meeting.

A paper entitled "Industrial Water and Water-Borne Industrial Waste," summarizing the activities of the committee, was prepared for publication in the ASTM BULLETIN.[1]

Manuscripts of all of the chapters to be included in the proposed ASTM Manual on Industrial Water have been submitted and are being edited.

A Proposed Guide for the Administration and Operation of Committee D-19 on Industrial Water has been completed.

The Chemical Section, Subcommittee on the Care of Pressure Vessels in Service, A.S.M.E. Boiler Code Committee, held no meetings in 1950. Max Hecht serves as the ASTM representative on both the Chemical Section and on the subcommittee. A conference group, comprising members of both the Chemical and Mechanical Sections, has prepared proposed revisions to the text of the 5th draft of Suggestions for the Care of Pressure Vessels in Service. Reference should be made to the 1950 report of the ASTM representative for the status of the work.[2]

The Executive Committee, Joint Research Committee on Boiler Feedwater studies held a regular meeting November 30, 1950, at New York, N. Y. R. C. Adams, vice-chairman of Committee D-19, was in attendance, substituting for the ASTM representatives, F. N. Speller and Max Hecht.

George E. Best of the Mutual Chemical Company of America, Baltimore, Md., has been appointed the liaison representative of the Research Committee, American Electroplaters' Society to Committee D-19 and to Subcommittee VII on Water-borne Industrial Waste. Committee D-19 has been requested to prepare standard methods based on the published research investigations sponsored by the A.E.S.

W. D. Hatfield, chairman, Committee on Standard Methods of Sewage and Industrial Waste Analysis of the Federation of Sewage and Industrial Wastes Associations, has been designated to serve as the liaison representative on Committee D-19 and Subcommittee VII on Water-borne Industrial Waste. L. Drew Betz, chairman of Subcommittee VII, has been appointed by the ASTM as the liaison representative to the F.S.I.W.A. Standard Methods Committee.

Frank N. Alquist was appointed the ASTM representative to the Research Council Engineering Foundation on Causes and Methods of Prevention of Internal Corrosion of Water Pipes.

At present, there are 110 members serving on Committee D-19, of whom 44 are classified as producers, 45 as consumers, and 27 as general interest members.

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* Presented at the Fifth-fourth Annual Meeting of the Society, June 18–22, 1951.

[1] ASTM BULLETIN, No. 161, September, 1950, p. 31.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 50, p. 399 (1950).

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1950 Annual Meeting, Committee D-19 presented to the Society through the Administrative Committee on Standards the proposed Tentative Specifications for Substitute Ocean Water. This recommendation was accepted by the Standards Committee on September 26, 1950, and the new tentative specifications appear in the 1950 Supplement to Book of ASTM Standards, Part 5, bearing the designation D 1141 - 50 T.

NEW TENTATIVES

The committee recommends for publication as tentative the following two methods, as appended hereto:[3]

Tentative Method of Test for Chloroform-Extractable Matter in Industrial Water.—This method describes a gravimetric procedure for the determination of chloroform-extractable matter in industrial water. See Table I for supporting data.

TABLE I.—EXPERIMENTAL DATA ON REPRODUCIBILITY OF THE PROPOSED TENTATIVE METHODS OF TEST FOR CHLOROFORM-EXTRACTABLE MATTER IN INDUSTRIAL WATER.

Mineral Oil added, ppm	Results obtained, ppm	Average, ppm	Difference, ppm	Percentage Difference
1.1	1.7 - 1.3	1.5	0.4	36
2.9	2.8 - 3.0	2.9	0.0	0
5.3	5.6 - 5.3	5.45	0.15	2.8
64	63	...	1.0	1.7
70	70	...	0.0	0
1007	990	...	17	1.6
1021	1008	...	13	1.2

Tentative Methods of Test for Fluoride Ion in Industrial Water.—These methods consist of a volumetric referee type procedure and a colorimetric non-referee type procedure for the determination of fluoride ion in industrial water.

REVISION OF TENTATIVE

Committee D-19 recommends that the *Tentative Methods of Test for Acidity and Alkalinity in Industrial Water* (**D 1067 - 49 T**) be revised as appended hereto.[4]

ADOPTION OF TENTATIVES AS STANDARDS

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standards, with suitable revisions in form and with a change in title, as indicated:

Tentative Method of Test for Iron Bacteria in Industrial Water (**D 932 - 47 T**).[5]—Add the words "and Water-Formed Deposits" to the title of this method.

Tentative Methods of Test for Sulfate-Reducing Bacteria in Industrial Water and Water-Formed Deposits (**D 993 - 48 T**).[5]

REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption the revision of Standard Definitions of Terms Relating to Industrial Water (**D 1129 - 50**),[6] expanding the definitions to include:

Industrial Water.—Water (including its impurities) used directly or indirectly in industrial processes.

Revise the definition of "Corrosion Products" to read as follows:

Corrosion Deposit.—A deposit which results from chemical or electrochemical reaction between a metal and its environment.

NOTE.—Corrosion deposits are usually precipitated on or near the corroded area. In certain instances, however, they may be deposited a considerable distance from the point at which the metal is undergoing attack.

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[3] The new tentatives were accepted by the Society and appear in the 1951 Supplement to Book of ASTM Standards, Part 5.

[4] The revised methods were accepted by the Society and appear in the 1951 Supplement to Book of ASTM Standards, Part 5.

[5] 1949 Book of ASTM Standards, Part 5.

[6] 1950 Supplement to ASTM Book of Standards, Part 5.

The committee asks for a nine-tenths affirmative vote at the Annual Meeting in order that this recommendation may be referred to letter ballot of the Society.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends that the following four tentatives be continued as tentative without revision, pending further study:

Tentative Method of:

Sampling Steam (D 1066 - 49 T).

Tentative Methods of Test for:

Dissolved Oxygen in Industrial Water (D 888 - 49 T),
Iron in Industrial Water (D 1068 - 49 T),
Suspended and Dissolved Solids in Industrial Water (D 1069 - 49 T).

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[7]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I, Advisory (Max Hecht, chairman).—The subcommittee held regular meetings, June 29 and October 16, 1950, at Atlantic City, N. J., and Pittsburgh, Pa., respectively. Proposals have been made to the executive officers of the Society seeking coordination of activities of national societies who prepare and publish methods of analysis for water. The objective of the proposals is to seek uniformity in standard methods. These proposals were studied at the June meeting. The members of the subcommittee endorsed the recommendation that Committee D-19 cooperate for the production of uniformity in standard methods for the analysis of water. This recommendation was subsequently approved by the members present at the committee meeting. It further approved the release for publication of the paper, "Industrial Water and Water-borne Industrial Waste,"[1] to foster such cooperation.

The marked increase in the committee membership in recent years and the ready acceptance by many to participate in the projects necessitated the preparation of a Guide for the Administration and Operation of the Committee and its distribution to all members of the Committee in February, 1951. The subjects covered by the Guide are as follows: scopes, organization, and membership; duties of elected officers and their appointees; preparation of proposed methods and revisions; processing of proposed methods and revisions; voting procedure; meetings, minutes, and reports.

M. D. Baker has been appointed as chairman of the Research Task Group of the subcommittee to prepare a report on research projects that the subcommittee wishes to refer to the society for information. The objective of the listing of the projects is to offer suggestions to technical schools for research investigations by students for thesis material or to industrial organizations for the solution of problems.

Subcommittee II, Editorial (Claude K. Rice, chairman) held a regular meeting on October 16, 1950, at Pittsburgh, Pa. At this meeting, the proposed "Recommendations on the Form of ASTM Methods and Specifications Relating to Industrial Water" was reviewed and approved on the third draft. It was further agreed to recommend this draft to Subcommittee I, Advisory, for inclusion as Part II of the "Proposed Guide for the Administration and Operation of ASTM Committee D-19 on Industrial Water."

The "Recommendations" referred to are supplementary to the Society's general "Recommendations on the Form of ASTM Standards" and are to be followed as closely as practicable, consistent with the purpose of the specified method of sampling, specific method of test, or specification being prepared.

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[7] The letter ballot vote on the recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

During the past year, this subcommittee has reviewed five proposed new and revised tentatives prior to their submittal to the Society, and also a proposed specification and a proposed revised method in draft form. Necessary editorial revisions have been made to put the proposed specifications and methods into the recommended form.

Section A on the ASTM Manual on Industrial Water has met twice in the past year. At the first meeting immediately after the 1950 Annual Meeting, June 30, 1950 at Philadelphia, Pa., the revised drafts of the individual chapters were reviewed in detail. This review disclosed a few omissions and inconsistencies among chapters. The missing material was supplied by chapter authors, and the chairman is now editing the chapters for consolidation into the Manual.

At the second meeting, March 8, 1951, at Cincinnati, Ohio, plans for illustrating the Manual and for preparation of the Glossary were agreed upon. The present schedule foresees distribution of draft copies of the assembled Manual to the section members this fall. In the light of their corrections or errors in the text, the chairman will then make a final editing and submit the complete text to the Society for publication.

Subcommittee III on Methods of Sampling (O. M. Elliott, Chairman).—During the past two years, the Tentative Method for Sampling Boiler Water (D 860 - 48 T) has been in the hands of the task group for substance revisions. It has now been revised, edited, and is ready for presentation to the committee for letter ballot to be continued as tentative.

The controversial matter of filtering boiler water samples at full boiler temperature was resolved by stating it in principle but not making it mandatory. Suitable trouble-free boiler temperature water filtering devices, which will remove phosphate sludge, are not available at present.

A draft of the Proposed Specifications for Equipment for Sampling Industrial Water and Steam has been prepared, distributed, and discussed by the task group. The fifth draft has been prepared in the format of the committee. This draft will be distributed to the committee for letter ballot vote, for recommendation to the Society for publication as tentative.

The subcommittee recommended that the Tentative Method of Steam Sampling (D 1066 - 49 T) be continued as tentative subject to further study.

Subcommittee IV on Methods of Analysis (F. E. Clarke, chairman).—The subcommittee held regular meetings on June 30, 1950, and March 9, 1951. It completed work on two methods of test for fluoride ions in industrial water and a method of test for chloroform extractable matters in industrial water, as appended to this report.[3]

Section A on Analytical Methods has completed work on the substance for proposed tentative methods for residual chlorine, iodide ion, copper ion and nitrite ion in industrial waters. The drafts of these methods, when editorially approved, are ready for letter ballot by the committee. Work is progressing constructively on proposed tentatives for clarity, turbidity and color; hydrogen; sulfide ion; bromide ion and master scheme of analysis for industrial water. Work on revisions of the Phosphate Method D 515 and Dissolved Oxygen Method D 888 is nearing completion. New projects have been started on proposed tentatives for total hardness by the modified Schwarzenbach titration method and for total carbon in industrial water.

Section B on Electrometric Methods has started work on a new proposed tentative for measurement of oxidation-reduction values in industrial waters and aqueous solutions.

Section C on Special Methods has completed the substance on a new proposed tentative for chemical microscopy as related to industrial waters and water-formed deposits. The draft, when editorially approved, will be distributed for letter ballot action by the committee. Work is progressing on proposed tentative methods for examination of water and water-formed deposits with the optical spectroscope and the flame photometer.

Section D on Chemical Analysis of Water-formed Deposits is preparing a final draft of a proposed tentative and plans to try it in a round robin test before circulating it for letter ballot.

Subcommittee V on Classification (S. K. Love, chairman).—The subcommittee held regular meetings on June 30, 1950, and March 9, 1951. The section on Nomenclature and Terminology submitted a definition for industrial water and a revision in the definition for corrosion deposit published elsewhere in this report. After slight changes, these definitions were approved by the subcommittee subject to letter ballot approval by Committee D-19.

The Specifications for Reagent Water was approved for substance and the draft, editorially approved, will be distributed to the committee for letter ballot.

Subcommittee VI on Methods of Testing (V. V. Kendall, chairman).—The subcommittee held regular meetings on June 30, 1950, and March 8, 1951. Specification for Substitute Ocean Water (D 1141 - 50) was approved by the committee by letter ballot in August and accepted for the Society by the Administrative Committee on Standards in September. Specification D 807 - 49 on the Embrittlement Detector is in process of review.

The Task Group on Service Testing of Tubular Products reported that a proposed specification for a miniature heat exchanger for studying the depository and corrosive characteristics of a water would be distributed to the subcommittee for comment.

Subcommittee VII on Water-Borne Industrial Wastes (L. D. Betz, chairman).—The subcommittee held regular meetings on June 29, 1950, and March 8, 1951.

Section A on The Critical Constituents of Water-Borne Industrial Wastes reported that all members had been asked to report on the critical constituents of the wastes from various industries. Since many industries are not represented by membership on this committee, additional groups are to be appointed to investigate and list the critical constituents of such other industries.

Section B on Methods of Analysis for Industrial Wastes has a total of 12 task groups engaged in developing methods of analysis. Proposed scopes for four methods have been submitted by this section for action and approval of Subcommittee I. These are: (*1*) Proposed Method of Test for Odor in Industrial Waste Water; (2) Proposed Method of Test for the Toxicity of Industrial Wastes to Fresh Water Fish; (*3*) Proposed Method of Test for the Chlorine Demand of Industrial Wastes; and (*4*) Proposed Method of Test for Dissolved Matter in Industrial Wastes. These scopes have been approved by Subcommittee VII.

Section C on Methods of Sampling, Gaging and Preservation of Samples has been actively continuing its work. One scope on "Methods for Gaging and

Sampling of Industrial Waste Waters" was submitted to and approved by Subcommittee VII and will be submitted to Subcommittee I for approval. Another scope, "Method for Preservations of Samples of Industrial Waste Waters" is complete except for wording and will be ready for approval at the next meeting of the Subcommittee. Three proposed procedures for flow measurement have been approved by the section and will be circulated to the subcommittee membership for further study and comments. The section solicits the assistance of Committee E-11 on the frequency and duration of sampling. Section C approved and presented to Subcommittee VII a request for research assistance through the Coordinating Subcommittee on Research to develop methods for preventing chemical, physical, and biological changes of samples which effect B.O.D. suspended solids, cyanides and phenol values. Containers for shipping refrigerated samples were also discussed by the sections.

Section D on Methods of Reporting Results of Analysis reported that questionnaires had been sent to the members of the section requesting information on the nature of their water-borne wastes, methods of analyses, and methods of reporting analytical results. This information should prove of value to not only Section D but also to Sections A and B. General agreement has been reached in Section D that, in reporting the analyses of water-borne industrial wastes, that reports should be in parts per million up to 10,000 ppm, after which weight per cent should be employed. Reports on taste and odor should be made employing "threshold value." It has also been agreed that temperatures should be expressed in degrees Centigrade.

The chairman also appointed a committee to assemble information and to make up a list of all associations and societies having committees and subcommittees on waste disposal. It is felt that this information will be of considerable value to prevent duplication of effort. Those organizations may have a common interest in methods of evaluating the constituents in industrial waste water.

This report has been submitted to letter ballot of Committee D-19, which consists of 109 members; 70 members have returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

MAX HECHT,
Chairman.

R. T. SHEEN,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-19 presented to the Society through the Administrative Committee on Standards the following recommendations:

Tentative Specifications for:

Equipment for the Sampling of Industrial Water and Steam (D 1192 - 51 T), and
Reagent Water (D 1193 - 51 T).

Revision of Tentative Method of:

Sampling Boiler Water from Stationary Boilers (D 860 - 48 T).

These recommendations were accepted by the Standards Committee on September 12, 1951, and the new specifications and revised method appear in the 1951 Supplement to Book of ASTM Standards, Part 5.

REPORT OF COMMITTEE D-20
ON
PLASTICS*

Committee D-20 on Plastics and its subcommittees held three meetings during the year: at Atlantic City, N. J., on June 28 to 30, 1950; in Cincinnati, Ohio, on November 14 and 15, 1950; and in Washington, D. C., on March 27 and 28, 1951. The Advisory Committee also met at these meetings.

It is with regret that we record the death of W. E. Emley on June 5, 1951. He was the first chairman of Committee D-20. He will long be remembered for his pioneer work with this committee.

This committee is participating in the International Standards Organization to promote international standardization in the plastic field. The secretariat for ISO/TC/61 on Plastics has been assumed by the USA, and Committee D-20 is being looked to to head up this work. A Special Subcommittee under the chairmanship of E. B. Cooper has been formed to handle the secretary's duties. The committee has held several meetings and has completed plans for a meeting to be held September 17 and 18 in New York City with representatives from the various participating nations who will be delegates. At this meeting it is expected that consideration will be given to three of the D-20 methods.

At the last meeting, a new subcommittee was formed to be known as Subcommittee XI on Editorial Review. It is expected that this new subcommittee will function shortly after the appointment of a chairman.

Committee D-20 will take an active part in the newly formed Committee on Cellulose and Cellulose Derivatives.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1950 Annual Meeting, Committee D-20 presented to the Society through the Administrative Committee on Standards the following recommendations:

Revision of Tentative Methods of:

Conditioning Plastics and Electrical Insulating Materials for Testing (D 618 – 49 T), and
Test for Repeated Flexural Stress (Fatigue) of Plastics (D 671 – 49 T).

Revision of Tentative Specifications for:

Cellulose Acetate Molding Compounds (D 706 - 48 T), and
Cellulose Acetate Butyrate Molding Compounds (D 707 – 47 T).

Revision and Reversion to Tentative of Standard Method for:

Long-Time Tension Tests of Plastics (D 674 - 48).

These recommendations were accepted by the Standards Committee on March 2, 1951 and appear in the 1951 Supplement to Book of ASTM Standards, Part 6.

RECOMMENDATIONS AFFECTING STANDARDS

The committee is presenting for publication three new tentatives and is recommending the revision of three tentatives, adoption as standard of six tentatives, revision of three standards for immediate adoption, and the withdrawal of one tentative. The standards and tentatives affected, together with the revisions recommended, are listed in detail in the Appendix.

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* Presented at the Fifty-fourth Annual Meeting of the Society, June 18-22, 1951.

The committee has considered all of the other tentatives, not mentioned in the Appendix, which are under its jurisdiction and which have been issued for two years or longer, and recommends that they be continued in their present status until further revisions in them are made as contemplated.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[1]

ACTIVITIES OF SUBCOMMITTEES

All of the subcommittees have been quite active during the past year. The Subcommittee on Permanence Properties has expanded its work, as has the Subcommittee on Specifications. There has always been a large attendance at the meetings of the Subcommittee on Research where a number of papers are presented.

Subcommittee I on Strength Properties (M. E. Marks, chairman).—Section A on Tensile and Compressive Strength is revising its method and the Section on Burst Strength is submitting a method for bursting strength of tubes.

The ski-ball test is still under consideration and a new method for determining impact strength will be submitted to the subcommittee soon after completion of a round-robin test. Since the Standard Method of Test for Stiffness in Flexure of Plastics (D 747 - 50) is being used widely by the industry at the present time, the Section on Flexural Strength is now considering a test for thin materials. This work is proceeding in conjunction with the efforts of Committee D-9 on Electrical Insulating Materials. The Section on Bond Strength is attempting to discover new adhesives which can be used for silicone laminates. A comparison study of various tear methods is being made by the Tear Strength Section in conjunction with the Society of Plastics Industry. During the past year a new Section on Dynamic Properties was formed and, at the present time, is compiling a list of methods used in the industry, as well as a list of definitions applicable to the subject.

Subcommittee II on Hardness (L. W. A. Meyer, chairman).—Extensive round-robin tests are now in progress and preliminary results will be reported shortly on abrasion resistance (optical effects) by the section. Also the Section on Abrasion Resistance (mechanical effects) has an additional round-robin test to complete.

The subcommittee at this time believes that the Tentative Method of Test for Resistance of Transparent Plastics to Surface Abrasion (D 1044 - 49 T) needs revising and therefore will remain tentative for the present. Work is still continuing on scratch resistance.

Subcommittee III on Thermal Properties (E. B. Cooper, chairman) has been quite active. Considerable work is in progress on flammability tests on thin plastic film, which project is being developed in cooperation with a committee of the Society of the Plastics Industry.

Round-robin testing will soon be organized in the Section on Flow of Thermoplastics, using commercial available models of the melt indexer for polyethylene, towards developing a test method. Further study of brittleness at low temperatures is being made by that section. Work is also progressing on post formability and some progress has been made towards developing a test method.

Subcommittee IV on Optical Properties (H. K. Hammond, 3rd, chairman).—A short time ago a questionnaire was circulated to members of Committee D-20 to determine the usefulness of present test methods and also to determine the desired direction for new activities within this subcommittee. As a result, the scope of Section A has been revised to include color and color difference which seem to be the fields of the greatest interest at present. A work program following this general scope is now being prepared.

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[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

The Tentative Method of Test for Haze of Transparent Plastics by Photoelectric Cell (D 672 – 45 T) is being withdrawn in favor of the Tentative Method of Test for Haze and Luminous Transmittance of Transparent Plastics (D 1003 – 49 T), which is being continued as a tentative method.

The subcommittee is studying the Tentative Method of Test for 60-deg Specular Gloss of Paint Finishes (D 523 – 48 T), prepared by Committee D-1 on Paint, Varnish, Lacquer, and Related Products. It has requested Committee D-1 to delete the phrase "of paint finishes" from the title to indicate its general applicability. The investigation of gloss methods is still being continued jointly with Committee D-1 and Committee E-12 on Appearance.

Subcommittee V on Permanence Properties (W. C. Goggin, chairman).—The Section on Effect of Light is studying means of obtaining a regulated 110-v, 60-cycle, single phase power supply for use with the S-1 sun lamp apparatus as used in the Standard Method of Test for Colorfastness of Plastics to Light (D 620 – 49).

A method on the resistance of plastic film to various liquids, as well as a revision of the Standard Method of Test for Resistance of Plastics to Chemical Reagents (D 543 – 43), will shortly be submitted to the subcommittee for ballot. One of the sections is completing a method of test for shrinkage of plastic film and sheets and a test for shrinkage of thermosetting plastics. Work on a test method for gas permeability is continuing.

Subcommittee VI on Specifications (Lucius Gilman, chairman) is preparing several new tentatives and revisions of a number of standard specifications. It is expected that a revision of the Tentative Specifications for Phenolic Molding Compounds (D 700 – 49 T) will be completed before the next annual meeting. The Section on Laminated Thermosetting Materials has been reorganized in view of the need for a specification on decorative laminates.

In cooperation with Committee D-9, the Tentative Specifications for Laminated Thermosetting Materials (D 709 – 49 T) are being extensively revised and enlarged. This revision covering laminated sheets, rods, and tubes, will result in uniform requirements to meet the industrial trade, and will be in agreement with the National Electrical Manufacturer's Association Standards as well as Military specifications.

Further study of the Tentative Specifications for Polystyrene Molding Compounds (D 703 - 49 T) will be made during the year, based on certain recommended changes. Specifications for nylon plastics, as well as for alkyd molding materials, are being prepared.

The Section on Cellulose Propionate Plastics has been abandoned since this molding powder has been taken off the market recently by the only company manufacturing this particular type of material.

A new section will be established to study the preparation of specifications for polyester resins, since specifications for this class of material have been prepared by the Government.

Subcommittee VII on Analytical Methods (D. E. Northrop, chairman).—A number of the sections have been quite active. Study is being made of a method for determining the amount of plasticizer. A method for testing all of the important analytical characteristics of vinyl plastic materials is under development. The Section on Plasticizers is still continuing its work on the Tentative Method of Sampling and Testing Plasticizer Used in Plastics (D 1045 – 49 T).

Work will be started on a method of testing polyesters when Subcommittee VI commences its work on this particular type of plastic.

Work on plasticizer analysis is being carried out jointly with Subcommittee XXV of Committee D-1.

Subcommittee VIII on Research (C. R. Stock, chairman) is still continuing to assist educational institutions in an advisory capacity on research problems. Work is still continuing on impact and flow properties of plastics.

At the November, 1950, Meeting of this subcommittee, the following two papers were presented:

"Thermal Behavior of Some Molded Thermoplastics," by R. M. Schulken, Tennessee Eastman Co.

"Mechanical Equation of State with Special Attention to Creep of Plastics," by W. N. Findley, University of Illinois.

The subcommittee also arranged, in conjunction with Subcommittee XII on Electrical Tests of Committee D-9, for presentation of the following six papers at the March, 1951, Meeting:

"Constant Deflection Stress-Relaxation Test for Plastics," by R. Burns and E. E. Wright, of the Bell Telephone Laboratories,

"Heat Distortion of Polystyrene," by K. J. Cleereman, J. L. Williams, and H. J. Karam, of the Dow Chemical Co.,

"A Simple Rheometer for Measuring Melt Flow of Polyethylene," by J. P. Tordella of E. I. duPont de Nemours Co., Inc.,

"Thermal Properties of Certain Laminated Plastics," by E. M. Schoenborn, A. A. Armstrong, Jr., and K. O. Beatty, Jr., of North Carolina State University,[2]

"Effect of Plastics Vapors on Electrical Arcing," by L. H. Germer, of the Bell Telephone Laboratories,

"A Rapid Test Method for Classifying Parallel and Perpendicular Electrical Quality of Mica Films," by K. G. Coutlee, of the Bell Telephone Laboratories.

Subcommittee IX on Molds and Molding Processes (J. L. Williams, chairman) has revised a number of its methods this year and also has made changes in the present molds to permit use of standard ASTM thermometers. The revisions are given in the Appendix.

Subcommittee X on Definitions, Nomenclature, and Significance of Test (C. H. Alexander, chairman) is still continuing its work on definitions for various terms relating to plastics.

The subcommittee has reviewed and approved certain definitions prepared by Committee D-14 on Adhesives, which also apply to plastics, as additions to the Tentative Definitions of Terms Relating to Plastics (D 883 – 49 T). Correlations of definitions with the special committee, appointed by Committee E-1 on Methods of Testing, and with Committee D-20 and other technical committees of the Society, is still continuing.

Subcommittee XIV on Conditioning (A. C. Webber, chairman).—Work is continuing on a new specification for servicing units and enclosures for test of plastics and electrical insulating materials at other than normal temperatures. Technical information on all methods of measurements of relative humidity is being obtained so the material can be organized and presented to this subcommittee as information. Any information along these lines will be welcomed by the subcommittee.

The work of this subcommittee is a joint function with Committee D-9.

This report has been submitted to letter ballot of the committee, which consists of 153 members; 66 members returned their ballots, of whom 60 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

G. M. KLINE,
Chairman.

B. L. LEWIS,
Secretary.

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[2] ASTM BULLETIN, No. 174, May, 1951, p. 54.

APPENDIX

RECOMMENDATIONS AFFECTING STANDARDS ON PLASTICS

In this Appendix are given a list of 3 new tentatives, proposed revisions in 3 standards and 3 tentatives covering plastics which are recommended as referred to earlier in this report. In addition, it includes recommendations for the adoption as standard of 6 tentatives and the withdrawal of 1 tentative. The tentatives and standards in their present form appear in the 1949 Book of ASTM Standards, Part 6, and its 1950 Supplement, as indicated by the final number in the ASTM designation.

New Tentatives

Committee D-20 recommends the following three proposed methods for publication as tentative by the Society as appended hereto.[1]

Tentative Method of Test for Bursting Strength of Round Rigid Plastic Tubing:

Since round plastic tubing is often subjected to internal hydrostatic pressure, this method is being recommended for publication.

Tentative Method of Test for Measuring Warpage in Sheet Plastics:

The committee is recommending this method for publication since there is a definite need for measuring warpage in sheet plastic specimens due to dimensional distortion resulting from exposure under service conditions.

Tentative Method of Test for Apparent Density and Bulk Factor of Granular Thermoplastic Molding Powder:

The committee is recommending this method for publication as a companion standard to the method for nonpouring molding powders (D 954).

Revisions of Tentatives

The committee recommends revisions in the following four tentatives:

Tentative Specifications for Melamine-Formaldehyde Molding Compounds (D 704 – 50 T):

In order to bring these specifications into agreement with present industrial practice, the following revision is recommended:

Table I. Change the flexural strength requirement for Type 6 from "13 000" to "11 000" psi.

In D 704 - 50T there is a typographical error and the value for flexural strength of Type 4 is printed as "1 000" psi instead of "13 000."

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[1] The new tentatives were accepted by the Society and appear in the 1951 Supplement to Book of ASTM Standards, Part 6.

Tentative Specifications for Molds for Test Specimens of Plastic Molding Materials (D 647 – 50 T):

A design of a compression mold for phenolic materials, as well as changes to permit use of more readily obtainable thermometers, are being recommended. The revisions are as follows:

Section 4.—Change to read as follows:

4. (*a*) *Molds for Tension Test Specimen.*—Molds for the test specimen for determining the tensile strength of molded electrical insulating materials (Note 1) shall be of the design and dimensions shown in Fig. 3.

(*b*) Molds for the test specimen for determining the tensile properties of plastics (Note 2) shall be of the design and dimensions shown in Fig. 8.

NOTE 1.—See Standard Method of Test for Tensile Strength of Molded Electrical Insulating Materials (ASTM Designation: D 654).

NOTE 2.—See Tentative Methods of Test for Tensile Properties of Plastics (ASTM Designation: D 638 – 49 T).

New Fig. 8.—Add the accompanying Fig. 1, referred to in Section 4(*b*) as Fig. 8.

Fig. 2—Mold for Disk Test Specimens.—Change the thermometer hole diameter from "$\frac{9}{32}$ in." to "$\frac{5}{16}$ in." Also revise the detailed drawings for the construction of molds to show a thermometer well diameter of "$\frac{5}{16}$ in." Add a note to Fig. 2 reading as follows: "NOTE.—Thermometer wells shall be $\frac{5}{16}$ in. in diameter to permit use of a readily available thermometer."

Tentative Definitions of Terms Relating to Plastics (D 883 – 49 T):

A number of new definitions, and also some definitions prepared by Committee D-14, that are applicable to plastic materials are recommended for inclusion in this revised tentative as appended hereto.[2]

ADOPTION OF TENTATIVES AS STANDARD

The committee recommends that the following seven tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Methods of Test for:

Deformation of Plastics under Load (D 621 – 48 T),

Rockwell Hardness of Plastics and Electrical Insulating Materials (D 785 – 48 T), jointly with Committee D-9,

Bond Strength of Plastics and Electrical Insulating Materials (D 952 – 48 T),

Measuring Changes in Linear Dimensions of Plastics (D 1042 – 49 T), and

Stiffness Properties of Nonrigid Plastics as a Function of Temperature by Means of a Torsional Test (D 1043 – 49 T).

Tentative Recommended Practices for:

Maintaining Constant Relative Humidity by Means of Aqueous Solutions (D 1041 – 49 T), jointly with Committee D-9.

REVISION OF STANDARDS, IMMEDIATE ADOPTION

Committee D-20 recommends that the following four standards be revised as indicated, and accordingly requests the necessary nine-tenths affirmative vote at the Annual Meeting in order that these recommendations may be referred to letter ballot of the Society:

Recommended Practice for Molding Specimens of Phenolic Materials (D 796 – 49), and Recommended Practice for Molding Specimens of Amino Plastics (D 956 – 50):

The committee recommends this revision to permit the use of available standard thermometers:

Section 2(d).—Revise to read as follows:

(*d*) *Thermometer.*—A 3-in. immersion, mercury-filled thermometer having a temperature scale of not more than 25 C (45 F) per inch of length, or a surface pyrometer graduated to read 2.5 C (5 F) shall be used.

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[2] The revised definitions were accepted by the Society and appear in the 1951 Supplement to Book of ASTM Standards, Part 6.

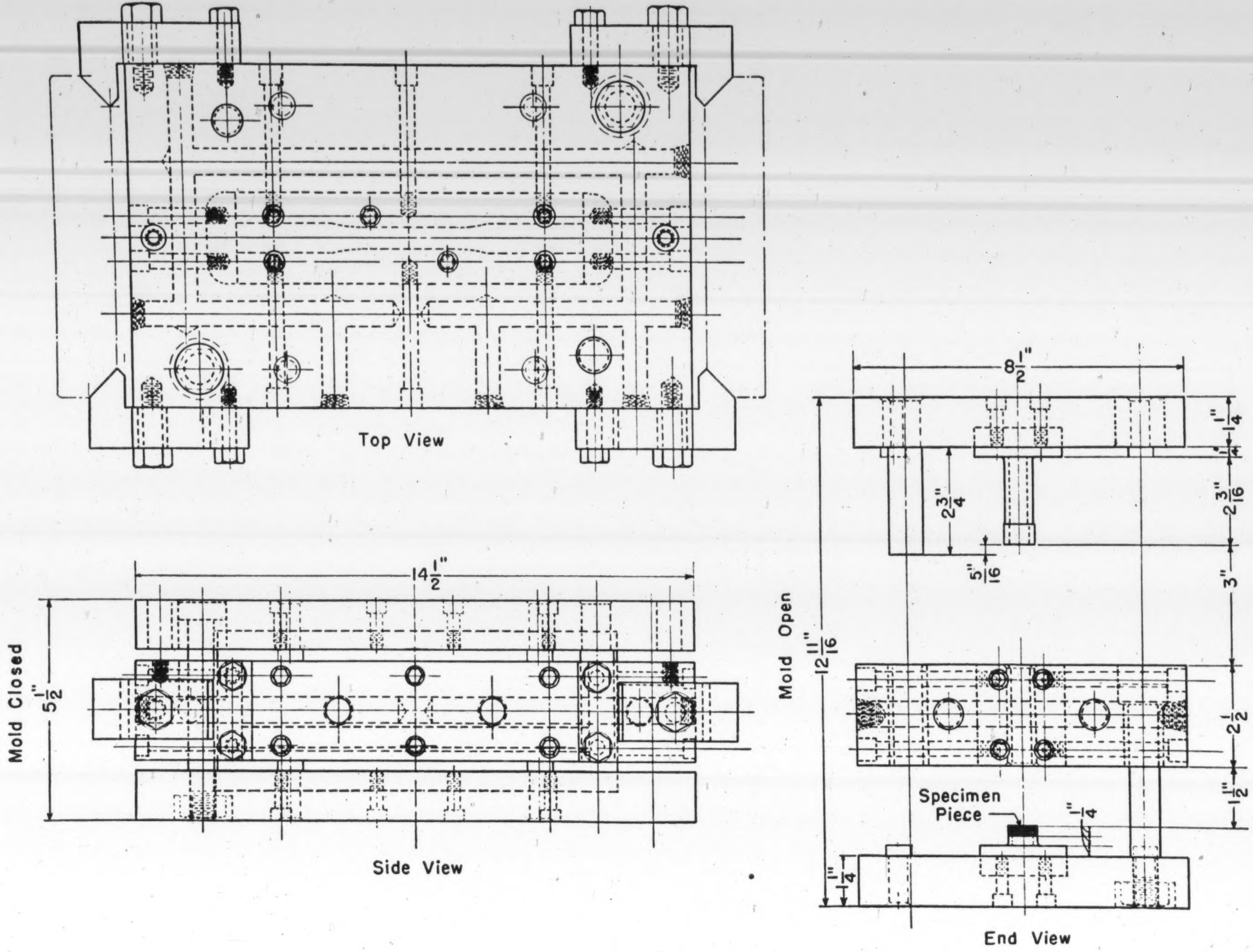
Top View
14 1/2"
Mold Closed
5 1/2"
Side View
8 1/2"
1/4"
2 3/16"
3"
2 1/2"
1 1/2"
2 3/4"
5/16"
Mold Open
12 11/16"
Specimen Piece
1/4"
1/4"
End View

FIG. 8.—Semi-Automatic Compression Mold for Molding Tension Test Specimens.

NOTE.—Thermometer wells shall be $\frac{4}{16}$ in. in diameter to permit use of a readily available thermometer.

Recommended Practice for Determining Temperatures of Standard ASTM Molds for Test Specimens of Plastics (D 958 – 50):

Revise Section 3 to read as follows:

3. *Thermometer.*—A 3-in. immersion, mercury-filled thermometer having a temperature scale of not more than 25 C (45 F) per inch of length shall be used.

Tentative Method of Measuring Shrinkage from Mold Dimensions of Molded Plastics (D 955 – 50):

This revision is being recommended to permit additional use of the 4-in. diameter by ⅛-in. disk for compression molded specimens. At present, only a ½ by ½ by 5-in. bar is permitted, while for injection molding, a ½ by ⅛ by 5-in. bar and a 4-in. diameter by ⅛-in. disk are permitted. The revisions are as follows:

Section 4(a).—Revise to read as follows by the addition of the italicized words:

4. (*a*) *Compression Mold.*—A single bar, single cavity positive mold ***and for diametral shrinkage, the single cavity positive 4-in. disk mold, both*** conforming to the Tentative Specifications for Molds for Test Specimens of Plastic Molding Materials (ASTM Designation: D 647).

Section 5(a).—Revise to read as follows by the addition of the italicized words:

5. (*a*) *Compression-Molding Materials.*—For mold shrinkages of compression-molding materials, the test specimens shall be bars, ½ by ½ by 5 in. ***or a disk ⅛ in. in thickness and 4 in. in diameter*** made in a positive mold in such a way that there is no appreciable lateral movement of the plastic during the molding.

WITHDRAWAL OF TENTATIVE

The committee recommends the withdrawal of the Tentative Method of Test for Haze of Transparent Plastics by Photoelectric Cell (D 672 – 45 T), which has been superseded by the Standard Method of Test for Haze and Luminous Transmittance of Transparent Plastics (D 1003 – 49 T).

REPORT OF COMMITTEE D-21

ON

WAX POLISHES AND RELATED MATERIALS*

Committee D-21 on Wax Polishes and Related Materials held two meetings during the year: the first in Washington, D. C., on September 14 and 15, 1950, and the second in Chicago, Ill., and Racine, Wis., on May 2 and 3, 1951. In addition, subcommittee meetings were held in New York City on December 6, 1950. All meetings were well attended and much work was accomplished. An interesting feature of the May meeting was an inspection trip through the recently completed Research Laboratories of S. C. Johnson and Son, Inc.

During the year the work of organizing the committee was completed and the following officers were elected:

Chairman, W. W. Walton.

Vice Chairman, J. Vernon Steinle.

Secretary, B. S. Johnson.

Additions and changes to the list of committee memberships have brought the total number of members to 45, of whom 17 are classified as producers, 11 as consumers, and 17 as general interest members.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Nomenclature (Donald B. Hanley, chairman) has been assigned the task of defining the terms used in the work of the committee. Among other things the group is working on an exact definition of the word "wax."

Subcommittee II on Raw Materials (Melvin Fuld, chairman) is completing a survey of existing ASTM test methods to determine their applicability to the work of Committee D-21. The subcommittee is also conducting a series of cooperative tests on methods for determining dirt and water content of vegetable waxes. In addition, the group is investigating means for determining additives to vegetable waxes by several methods, including displacement chromatography.

Subcommittee III on Chemical and Physical Testing (Donald M. King, chairman) has been assigned the problem of developing test methods for the chemical and physical properties of wax polishes, prior to their use or application. Currently three cooperative programs are being conducted on methods for determining non-volatile matter, ash content, and free alkali in water-emulsion waxes.

Subcommittee IV on Performance Tests (Michael Sveda, chairman) is working on the development of procedures for testing the performance of wax polishes starting with the application of the finished products for final use. In order to facilitate its work, the subcommittee has been divided into three sections.

Section A on Slip Resistance is studying methods and apparatus for determining the slip resistance of waxed floors. A cooperative test program is in progress to investigate the usefulness of the Sigler Pendulum Impact machine and the Underwriters Laboratory machine for this purpose. As this work progresses, an effort will be made to correlate the laboratory results on waxed surfaces with actual slipperiness, as determined by field experience.

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*Presented at the Fifty-fourth Annual Meeting of the Society, June 18–22, 1951.

Section B on Service Life is working on the development of test methods for some of the many factors which influence wear resistance. It is felt that no single test can be developed which will give a direct measure of the service life of wax polishes. Cooperative test programs have been started on testing water spot resistance, flexibility, and adhesion of wax films so that the effect of these properties on service life can be evaluated. A method for measuring the hardness of wax films is also under discussion and a project may soon be started on this property.

Section C on Appearance is currently engaged in investigating methods of application of wax polishes. Because of the thin films involved, it is difficult to achieve uniform application on test areas so that the appearance factors can be measured quantitatively. Several methods are under discussion, and a cooperative test program has been set up to determine their relative merits.

Subcommittee V on Specifications (W. H. Joy, chairman) is greatly hampered in its work by the lack of test methods for many of the important properties of wax polishes. This situation should improve as the work of the other subcommittees progresses. At the present time this group is gathering together existing specifications for industrial-type water-emulsion waxes for study and review. While it is known that many of these specifications have proven inadequate, it is hoped that a study of the various chemical, physical, and performance tests included in them will lead to a better knowledge of the type of tests which should be included in a specification. Special emphasis is being placed on determining the significance of the various tests. A task force has also been assigned to investigate the possibility of preparing recommended practices for the preparation and waxing of floors.

This report has been submitted to letter ballot of the committee, which consists of 44 voting members; 33 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

W. W. Walton,
Chairman.

B. S. Johnson,
Secretary.

REPORT OF COMMITTEE E-1
ON
METHODS OF TESTING*

Committee E-1 on Methods of Testing held two meetings during the year at ASTM Headquarters in Philadelphia as follows: on November 10, 1950, and on May 18, 1951.

During ASTM Committee Week in Cincinnati, Ohio, on March 5 to March 9, 1951, meetings were held of six E-1 subcommittees and three task groups. Arrangements have been made for meetings of thirteen subcommittees and five task groups during the 1951 Annual Meeting of the Society.

The Board of Directors has appointed Messrs. D. E. Parsons, R. L. Templin and L. H. Winkler, each for another term of three years as members of Committee E-1.

A Task Group on Porous Filtering Bodies was organized on February 21, 1951, at a meeting in Washington, D. C., under the chairmanship of T. L. King, of Merck and Co. Consideration will be given to performance requirements for these types of filters, made of sintered glass, stainless steel, ceramicware, platinum sponge, etc.

A new Task Group D on Shear and Torsion Tests, under the chairmanship of F. S. Mapes, General Electric Co., was organized at a meeting held on March 6 in Cincinnati. Plans were made to prepare a bibliography, and a questionnaire will be circulated to members of the task group and others interested in this subject in order to obtain information regarding those phases of the subject that should be given first consideration.

In view of the wide interest in deep-drawing and forming properties of metals, a Study Group on Cupping Tests, under the chairmanship of E. F. Lundeen, was appointed. A meeting of this study group will be held during the 1951 Annual Meeting of the Society.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1950 Annual Meeting, Committee E-1 presented to the Society through the Administrative Committee on Standards the following recommendations:

Tentative Revision of Standard Specifications for:

ASTM Thermometers (E 1 - 50).

Revision and Reversion to Tentative of Standard Method of:

Test for Brinell Hardness of Metallic Materials (E 10 - 27).

These recommendations were accepted by the Standards Committee on September 26, 1950; the tentative revision of E-1 appears in Parts 3, 4, 5 and 6 of the 1950 Supplement to Book of ASTM Standards, and Tentative Method E-10 appears in Parts 1 and 2 of the 1950 Supplement.

REVISIONS OF TENTATIVES

Committee E-1 recommends revisions in the following four tentatives:

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* Presented at the Fifty-fourth Annual Meeting of the Society, June 18-22, 1951.

Tentative Methods of Compression Testing of Metallic Materials in Other Than Sheet Form (E 9 - 49 T),[1]

Tentative Method of Compression Testing of Metallic Materials in Sheet Form (E 78 - 49 T),[1]

Tentative Recommended Practice for Analysis by Microscopical Methods for Particle Size Distribution of Particulate Substances of Subsieve Size (E 20 - 48 T),[2] revision of Section 1 (*d*) and

Tentative Method of Test for Softening Point (Ball and Shouldered Ring Apparatus) (E 28 - 42 T).[3]

TABLE I.—PROPOSED REVISIONS IN THERMOMETER SPECIFICATIONS.

DIMENSIONS OF SCALES

Thermometer	Present Specification	Change Suggested
17C..............	135-150 mm to 19 C 20-35 mm to 27 C	136-151 mm bottom to 19 C 20-35 mm top to 26.7 C
18C..............	135-150 mm to 34 C 20-35 mm to 42 C	135-150 mm bottom to 34.4 C 22-37 mm top to 42 C
19C..............	135-150 mm to 49 C 20-35 mm to 57 C	136-151 mm bottom to 49 C 20-35 mm top to 56.7 C
20C..............	135-150 mm to 57 C 20-35 mm to 65 C	138-153 mm bottom to 57 C 20-35 mm top to 64.4 C
21C..............	135-150 mm to 79 C 20-35 mm to 87 C	136-151 mm bottom to 79 C 20-35 mm top to 86.7 C
22C..............	135-150 mm to 95 C 20-35 mm to 103 C	135-150 mm bottom to 95.6 C 23-38 mm top to 103 C

DIMENSIONS OF ENLARGEMENTS

Thermometer	Stem Diameter, mm	Diameter, mm	Length, mm	Bottom of Bulb to Bottom of Enlargement, mm
9C, 9F, 10C, 10F, 57C, and 57F...........	6.0 to 7.0	7.5 to 8.0	3.0 to 5.0	65
32C.....................................	6.0 to 7.0	8.0 to 10.0	4.0 to 7.0	150
17C, 17F, 18C, 18F 19C, 19F, 20C, 20F 21C, 21F, 22C, 22F	6.0 to 7.0	8.0 to 10.0	4.0 to 7.0	114

REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption the following revisions in the Standard Specifications for ASTM Thermometers (E 1 - 50) and accordingly requests the necessary nine-tenths affirmative vote at the Annual Meeting in order that this recommendation may be referred to letter ballot of the Society:

Section 6 (a).—Add the following at the end of the first sentence "The width of graduation lines shall be not more than one-fifth the graduation interval."

Section 8.—Revise Section 8 to read as follows:

8. The thermometer shall be supplied in a suitable case on which shall appear the following marking (except when a transparent case is used): the letters "ASTM," the thermometer number (33C, 33F, etc.), the name of the thermometer, and the temperature range.

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[1] The revised methods were accepted by the Society and appear in the 1951 Supplement to Book of ASTM Standards, Parts 1 and 2.

[2] The revised recommended practice was accepted by the Society and appears in the 1951 Supplement to Book of ASTM Standards, Parts 3 and 4.

[3] The revised method was accepted by the Society and appears in the 1951 Supplement to Book of ASTM Standards, Parts 3, 4, 5, and 6.

Table I.—Revise the Tank High Thermometer 60F to require the stem to be of the lens front type. Also make the changes in the thermometers indicated in Table I as regards the specified dimensioning of scales and enlargements.

REVISION OF STANDARD AND REVERSION TO TENTATIVE

The committee recommends the revision of Standard Methods of Tension Testing of Metallic Materials (E 8 - 46) and its reversion to tentative status, as appended hereto.[1]

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

ACTIVITIES OF SUBCOMMITTEES AND TASK GROUPS

While the subcommittees and task groups have been quite active during the year, the following summary covers briefly only those projects on which definite progress has been made or completed during the past year.

Subcommittee 2 on Effect of Speed in Mechanical Testing (P. G. McVetty, chairman) completed a revision of Section 20 on Speed of Testing which has been included in the revised Methods of Tension Testing of Metallic Materials (E 8 - 46).

Subcommittee 3 on Elastic Strength of Materials (F. M. Howell, chairman).—A Task Group is making a survey of work done in determining the elastic constants of materials and the need for information on elastic constants. It has prepared and distributed a questionnaire to about 100 commercial firms and laboratories and is now engaged in correlating the replies. If there is a need for methods of determining the elastic moduli, recommendations on suitable methods applicable to both metals and non-metals will be prepared.

Another Task Group is endeavoring to correlate the terminology and definitions pertaining to testing of both metals and non-metallic materials.

Subcommittee 4 on Tension Testing (R. L. Templin, chairman) completed the proposed revision of Standard Methods E 8, appended hereto,[2] which incorporates detailed testing procedures for testing steel spring wire and copper rods and bars, in addition to the procedures covered by the present methods. The revised methods represent the first step in a complete revision of Standard Methods E 8. During the year detailed consideration will be given to other features of the method, in order to bring them into agreement with Government specifications and recently issued standard of the British Standards Institute, and the Canadian Bureau of Mines. Study is also being made of recommendations received from Committee A-1 on steel. Consideration is being given to revision of the definitions of the terms "yield strength" and "yield point."

Work is continuing on a test for determining the bursting strength of thin metallic materials.

Subcommittee 6 on Indentation Hardness (R. H. Heyer, chairman).—In cooperation with Committee E-4 on Metallography, a proposed method of test for diamond pyramid hardness of metallic materials has been completed. This method covers test loads from 1 g to 120 kg using the square-based "Vickers" type indenter. It includes both the microhardness and the heavy load DPH tests. Micro-hardness tests using the Knoop indenter are not included. A Standard Hardness Conversion Table for Nickel and High-Nickel Alloys has also been completed. These two recommendations will be submitted as tentative to the Society later in the year.

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[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Task groups are studying the need for test methods for the rapid, direct reading types of hardness testers using the Brinell indenter, and for test methods for portable hardness testers.

Subcommittee 9 on Rheological Properties (W. F. Fair, Jr., chairman) completed the Report on the Principles Involved in the Determination of Absolute Viscosity, edited by W. H. Markwood, Jr., which appears as an Appendix to this report.

The Task Group on Softening Point completed the proposed revised Tentative Method of Test for Softening Point by Ring and Ball Method, appended hereto,[3] which represents a proposed consolidation of Methods E 28 and D 36. In cooperation with Committees D-1 and D-4 and other interested committees, further cooperative round-robin tests using the revised method will be made during the year. Any final changes resulting from these further studies will be made in the method next year, at which time it is hoped that the method will be ready for adoption as standard.

The Task Group on Saybolt Furol Viscosity of Asphaltic Materials at High Temperatures has completed further round-robin tests. Reproducibility has been quite good on relatively low-viscosity asphalts but very poor on high-viscosity air-blown asphalts. The details of the equipment and procedure are being further scrutinized and modified before undertaking additional cooperative work.

Subcommittee 14 on Conditioning and Weathering (A. C. Webber, chairman) has been requested to give consideration to the establishment of humidity testing conditions such as those encountered in tropical climates involving very high or saturated conditions. Plans are well under way for a Symposium on Conditioning and Weathering which will be sponsored by the committee at the 1952 Annual Meeting of the Society.

Subcommittee 16 on Thickness Measurement (K. G. Coutlee, chairman) is reactivating its program of work. It has received a request from Committee B-4 to consider thickness test procedures for certain cathode ray and radio tube parts.

Subcommittee 17 on Thermometers (R. M. Wilhelm, chairman) prepared the revisions in the Standard Specifications for ASTM Thermometers (E 1 – 50) presented earlier in this report. It has in preparation specifications for three new ASTM Kinematic Viscosity Thermometers and a new ASTM Oil in Wax Thermometer, all requested by Committee D-2.

Subcommittee 21 on Metalware Laboratory Apparatus (E. L. Ruh, chairman).—In cooperation with Research Division VIII of Committee D-2, recommendations have been made for the Tag closed and Pensky-Martens flash testers which involve adoption of a suitable ferrule to permit interchangeability of thermometers used in these two methods and the establishment of a testing gage for obtaining desired uniformity of thermometers.

A task group has been established to study laboratory ovens. It is hoped that standard specifications for various types of ovens will ultimately result from this work. Consideration is being given to analytical balances and laboratory gas burners. The attention of the subcommittee has been directed to certain existing specifications for metalware apparatus where errors exist and are causing trouble. After study of these cases, recommendations for appropriate revisions will be made to the technical committee responsible for the standards involved.

Task Group A on Distillation Tests (H. P. Ferguson, chairman) is endeavoring to find the best solution to the confusion now existing in determining the boiling range of various hydrocarbons. This problem arises from the desires of producers and consumers to standardize in accordance with their own industry tradition.

As an example, D. C. naphtha is furnished by the petroleum industry to both the paint and dry-cleaning industries. The petroleum industry prefers Distillation Method D 86 which is used for all petroleum products. The paint industry needs a more accurate method for other very narrow cut materials which it uses. The tendency, therefore, is to test D. C. naphtha by Method D 1078 rather than D 86.

The cooperation of the technical committees with the task group has been excellent. Committees D-1 and D-16 have almost eliminated all equipment differences between Methods D 850 and D 1078.

Committees D-1 and D-2 have appointed members to a Joint Committee to explore the best application of methods to the various products coming within their respective fields.

Task Group B on Low-Temperature Testing of Elastomers and Plastics (R. S. Havenhill, chairman).—Through the cooperative efforts of Mr. L. Boor and Mr. W. E. Scoville, Jr., the final results of the questionnaire sent out last year to a large number of laboratories have been tabulated and the results reported. The results, which represent 40 per cent returns and a good cross-section of industry, government and research centers, indicate that a majority of the laboratories are already standardized on ASTM low-temperature test methods. The survey, however, indicated the need for further correlation of test methods measuring the same basic properties of rubber and further standardization of tests. The replies to the questionnaire showed the following four tests to be those most used of the 100 tests reported upon for the evaluation of stocks for low-temperature service:

	Frequency of Use
1. Stiffness	67
2. Brittleness	54
3. Hardness	45
4. Rheological Properties—Compression, Set, Etc.	45

A section has been appointed to correlate the various methods of measuring these four basic properties.

Task Group C on Water Vapor Permeability (R. Burns, chairman).—A draft of a Proposed Method for Measuring Water Vapor Transmission was submitted on January 15, 1951, to members of Task Group C and to the officers of the interested technical committees. While the returns of the letter ballot indicated general approval, there were many comments on minor details which have necessitated a further revision in order to reconcile the viewpoints of the many committees involved. A new draft is being prepared and will be submitted to the technical committees for formal approval and shortly thereafter will be submitted to Committee E-1.

This report has been submitted to letter ballot of the committee, which consists of 12 members; 12 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

J. R. TOWNSEND,
Chairman.

P. J. SMITH,
Secretary.

APPENDIX

REPORT ON THE PRINCIPLES INVOLVED IN THE DETERMINATION OF ABSOLUTE VISCOSITY

EDITED BY W. H. MARKWOOD, JR.[1]

The Task Group on "Methods and Apparatus for Absolute Viscosity Measurements" of Subcommittee 9 on Rheological Properties was appointed by Committee E-1 on Methods of Testing to prepare a report containing simplified discussions of the various principles by which viscosity may be determined in absolute, as contrary to arbitrary, units and to describe several viscometers that illustrate these principles.

The purpose of the work is to encourage the recognition and practice of the use of instruments yielding results in standardized units having true physical dimensions that will convey the same meaning in all laboratories.

The report is not to be considered a laboratory manual, rather an introduction to rheology from a practical, schematic and phenomenological point of view while resting on a sound theoretical base.

The Task Group wishes to acknowledge its indebtedness to the following persons who have been generous contributors to the report: H. R. Beck, Mellon Institute of Industrial Research, Pittsburgh, Pa.; F. D. Dexter, Bakelite Corp., Boundbrook, N. J.; W. P. Mason, Bell Telephone Laboratories, Murray Hill, N. J.; H. Raich, Mellon Institute of Industrial Research, Pittsburgh, Pa.; T. L. Smith, Hercules Experiment Station, Wilmington, Del.; R. N. Traxler, The Texas Company, Port Neches, Tex.

W. F. Fair, Jr., *Chairman*
W. H. Markwood, Jr., *Secretary*
J. C. Geniesse
J. A. Murray
E. O. Rhodes
R. H. Sawyer
R. N. Traxler

Many viscometric methods involving a number of principles of motion may be found in the literature. They all serve in one way or another to describe the resistance of fluids to flow when acted upon by external forces. These instruments may be divided into two general classes: (1) those in which fluid motion may be interpreted in terms of fundamental viscometric units, dyne seconds per square centimeter or poises; and (2) those in which fluid motion is described in an arbitrary manner.

The intended purpose of this collaboration is to discuss methods belonging in the first group. It is not intended, however, to completely exhaust this field but to deal with instruments having wide acceptance. A further limiting factor will be consideration of the accessibility of the methods to all industries interested in the phenomena of fluid flow.

The means of determining viscosities in fundamental units can likewise be subdivided into (1) absolute viscometers, and (2) relative viscometers. The former allow the determination of viscosity without a knowledge of the viscosity of other fluids. The latter are useful only when "standard" materials of known viscosity are available for calibration purposes. This report will make no attempt to enter the controversy regarding the assignment of viscometers to one or the other of these categories. Both are useful for the determination of "absolute" viscosity, which is the matter of interest being reported here.

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[1] Hercules Experiment Station, Wilmington, Del.

The Contemporary Rheological Range:

Present day industry deals with practical problems involving fluid materials having viscosities ranging from small fractions of a poise up to billions of poises. Typical examples and the orders of magnitude of their viscosities are: hydrogen, 10^{-4} poises; liquid air, 10^{-3}; water and alcohol, 10^{-2}; lubricating oils, inks, varnishes, and paints, 10^0 to 10^3; fats and greases, 10 to 10^6; resins and gums, 10^3 to 10^9; pitches, asphalts, and plastics, 10^6 to 10^{12} and higher.

The rheological range is not only one of tremendous degree, it is also concerned with kinds of flow. The viscosities of some liquids do not change with changing rates of flow, others become more viscous as flow increases, still others become more fluid with increasing velocity of flow. Materials behaving in any of the above ways may or may not flow upon application of forces below certain limits.

These ranges of degree and kind have led to the development of numerous ways of observing the behavior of fluids in motion.

Viscometer Types:

Viscometers might be classified in several ways. One means of distinguishing between them is to group them generally according to the measurements employed: (1) The rate of fluid flow in tubes, (2) the rate of motion of solids through subject media, (3) the rate of rotation of solids in the media, (4) the distortion of solids by forces transmitted through the media from other solids in motion, (5) the damping effect of a medium on a solid vibrating in it, and (6) the distortion of media under compression. Examples of these types are:

1. Bingham Absolute Viscometer,
   Bingham Plastometer,
   Koppers Capillary Rise Viscometer.
2. Falling Sphere Viscometer,
   Hoeppler Rolling Sphere Viscometer,
   Falling Coaxial Cylinder Viscometer.
3. Stormer Rotating Cylinder Viscometer,
   Open End Rotating Cylinder Viscometer.
4. Interchemical Rotating Torsional Viscometer,
   MacMichael Rotating Torsional Viscometer.
5. Electromechanical Transducer.
6. Parallel Plate Plastometer.

The first example in group 1 employs measurement of the time required to transfer a known volume of sample from one part of the apparatus through a tube to another part. The time required to discharge a measured amount of sample through a capillary exit is determined with the second instrument. The time required to fill a measured portion of a capillary is observed with the third apparatus.

The rate of fall of a sphere a known distance through a liquid in a vertical cylinder is the effect observed with the first apparatus in group 2. A similar measurement is also made with the second viscometer but in this case the moving member is a sphere which rolls through a close-fitting, inclined tube containing the subject fluid. The rate at which one cylinder will fall out of another from which it is separated by the sample is observed in the third method.

The speed with which one cylinder will rotate inside another containing the sample, when a known force is applied, is the operating principle of the two examples given for group 3.

The sample container is rotated in the instruments listed in group 4. The torsion produced in a wire supporting a cylinder submerged in the liquid is measured when instrument one is used. The second apparatus differs from the first in that the cylinder may be replaced by a horizontally suspended disk.

The reaction of an electrically vibrated cylinder immersed in the sample is observed with the method listed in group 5.

The method listed in group 6 measures the motion of a sample compressed between two parallel plates.

The analysis and interpretation of the experimental results obtained with the aid of the various viscometers in these six classes differ appreciably and will be outlined briefly in the discussions of the individual instruments. Mathematical details will be omitted from these discussions for practical reasons. These details can be found by reference to the works cited in the bibliography and, in most cases, to any of the standard monographs on viscometry.

Before the viscometers are discussed separately, it might be well to review some of the underlying principles that govern all of them, that is, the relationships between force and flow that make up the study of the rheology of liquids.

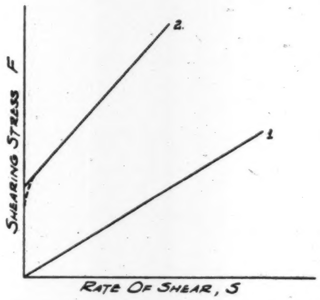

FIG. 1.—Relationship Between Applied Stress and Rate of Shear.

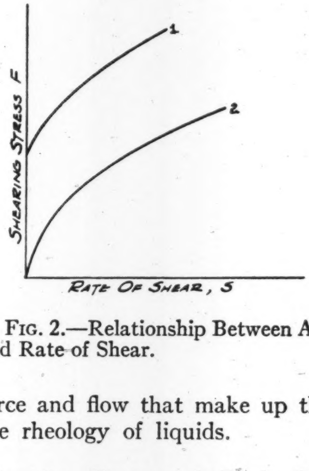

FIG. 2.—Relationship Between Applied Stress and Rate of Shear.

Viscosity—Stress and Strain Relationships:

Phenomena involving the deformation of matter are usually described in terms of the relationship existing between a force, called stress, producing motion within a material, and the amount of motion produced. A number of these phenomena are influenced also by the length of time required to produce this amount. These motions represent changes in shape that may involve lengths, areas, or volumes, or all of them, and the changes in shape are called strains. A kind of strain that occurs when areas slide past one another without being accompanied by changes in volume is called shear. The ratio of the velocity with which one of these areas slides past another to the distance they are apart is called rate of shear.

When a force is applied to one or more, but not all, surfaces of a body of liquid its constituent parts move, not as a continuum but with continually changing shape. That is, the distances between the constituent particles in any two planes is constantly changing. The relationship between the applied stress and the resultant rate of shear is not the same for all liquids, being quite simple in some cases and complex in others. It has been found that if this relationship is illustrated graphically curves of various types will be observed. Figures 1 and 2 illustrate some of the more fundamental ones. It is evident that they describe different kinds of fluid flow. The simplest (1) shows the rate of shear not only as a linear function of the shearing stress but also as being directly proportional to it. This type of flow diagram illustrates the behavior of a large class of fluids called "Newtonian" and is largely comprised of those having only one component such as water, alcohol, glycerin, benzene, etc.; however a large number of solutions also belong in this class. Although the relationships for these liquids are linear and pass through the origin of the stress and strain axes, the slopes of the lines may be quite different for different liquids. These slopes, or constants of proportionality, represented by the ratios of the shearing stresses to the rates of shear, are known as "coefficients of viscosity," or more simply, the "viscosities" of the liquids.

$$\eta S = F \ldots\ldots\ldots\ldots\ldots (1)$$

where:

S = rate of shear,
F = shearing stress, and
η = viscosity

The proportionality coefficient, η, is not a dimensionless number but is expressed by a compound unit, called the "poise," involving mass, distance, and time, since (if M = mass, L = distance or length and T = time),

$$S = \text{velocity/distance} = (L/T)/L = 1/T$$
$$F = \text{force per unit area} = (ML/T^2)/L^2 = M/LT^2$$
$$\text{and} \quad F/S = (M/LT^2)/(1/T) = M/LT = \eta$$

Curve 2 illustrates a kind of flow generally called "pseudo-viscous" (originally called "plastic"), though some object to the term. Again the stress, rate of strain relation, is linear, but in this case an intercept is seen on the stress axis. This is the force that must be applied before any flow of the material begins. This type of flow was first discussed in detail by Bingham and from his studies arose the well-known "Bingham Law of 'Plastic' Flow" which explained why the rate of shear of some liquids was not proportional to the stress applied to them

$$S = \mu(F - f) \tag{2}$$

where μ is the constant of proportionality and is represented by the slope opposite to that of the first class above, that is,

$$S/(F - f)$$

instead of F/S. Bingham called this slope the "mobility." It is comparable to the "fluidity" ($1/\eta$) of Newtonian liquids. In this relation f is the yield value, or stress intercept, that must first be subtracted from the stress at any shearing rate before the direct proportion can be expressed. It, like μ, is a property of the liquid. (It is probably proper to speak of $(F - f)/S$ as the "pseudo-viscosity" of such materials.) In some cases, possibly all, the relationship is not linear at very low shearing rates, so that the intercept is really less than f (possibly zero). Nevertheless, f is the value required to express the direct proportion and the linear part of the curve is the one usually observed in the practical range of measurement. Clay and pigment suspensions are examples of this class.

The behavior of still other materials is shown on Fig. 2, curve 1. This illustrates the kind of flow that is generally called "plastic" and is characterized not only by a yield value but also a steadily decreasing apparent viscosity, $(F - f)/S$, as the stress is increased. The proportionality constant, μ', in such cases has been described as an exponential function of F, f, and S

$$S = \mu'(F - f)^n \tag{3}$$

wherein n is the slope of a logarithmic plot of S *versus* $(F - f)$. When n is one, μ' becomes Bingham's mobility, but for any other value of n, μ' has little physical significance because its compound unit, involving mass, distance and time, may become meaningless. It would probably be best to describe materials exhibiting this kind of flow by reporting values for the apparent viscosity $(F - f)/S$ and the rates of shear at which they were observed.

Fig. 2, curve 2 is similar to Fig. 2, curve 1 except for the absence of a yield value. Liquids exhibiting this type of flow have been described as the "non-Newtonian" liquids. It does not seem advisable to limit this term to this type of liquid only. Rather, it should be applied generally to all liquids differing in flow character from those illustrated by curve 1, Fig. 1; if the latter are Newtonian, then all others are non-Newtonian. The Fig. 2, curve 2 type of flow has also been called "pseudo-plastic" which is logical if one considers that the absence of a yield value might be overlooked unless measurements were made at sufficiently low rates of shear. This kind of flow can be expressed by the "plastic" Eq. 3 wherein f is zero. It can then be specified by apparent viscosity values, F/S, and the rates of shear at which they were observed.

Although this discussion has by no means covered the field of anomalous viscosity, it has pointed to phenomena that should be borne in mind while making measurements of "absolute viscosity."

CAPILLARY VISCOMETERS

Observations of the flow of liquids through capillary tubes led Poiseuille (**1**)[1] to the conclusion that the volume of flow per unit time was directly proportional to the pressure drop through the tube and the fourth power of the capillary diameter, and inversely proportional to its length. The development of the mathematical relationships between pressure, volume of flow in unit time, shearing stress, rate of shear, and viscosity was probably first performed by Stokes (**2**) although the first published deduction of Poiseuille's observations was made by Wiedemann (**3**) and further developed by Hagenbach (**4**). The latter showed that

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[1] The boldface numbers in parentheses refer to the list of references appended to this report, see p. 472.

$$\eta = \frac{\pi R^4 Pt}{8LV} \quad \ldots\ldots\ldots\ldots (4)$$

wherein R is the radius of a capillary of length L through which will flow a volume V of liquid in a time t under the influence of a pressure drop P. For any one instrument R, V, and L are constant so that

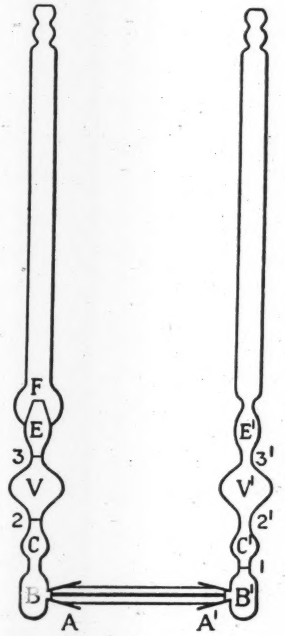

FIG. 3.—Bingham Absolute Viscometer.

$$\eta = KPt = k_1P/k_2(1/t) \quad \ldots\ldots\ldots (5)$$

wherein k_1P is the shearing stress and $k_2(1/t)$ is the rate of shear. Therefore, graphs of P *versus* $1/t$ should be representative of materials behaving as in Fig. 1, curve, 1. Careful observations showed that this was not always true and it was soon recognized that corrections for both P and L were required. Not all the force represented by P is employed to overcome viscous friction; part is used for the bodily transfer of the liquid particles from one place to another, that is, to impart kinetic energy. Furthermore, the flow in short tubes was found to be proportional not to L but to $L + \Delta L$, a "fictitious" length wherein ΔL is a function of R. This correction is the result of the rapidly changing velocities at the capillary entrance and exit. For capillaries in which L/R is greater than about 130, ΔL has been considered negligible (**5**). However, the "kinetic energy effect" may be quite appreciable at other than very low velocities. A number of investigations (**6**) have shown this to be a function of liquid density (ρ), volume flow rate, and capillary radius

$$p_k = m\rho(V/t)^2/\pi^2R^4 \quad \ldots\ldots\ldots (6)$$

wherein m is a proportionality constant. Various values have been calculated for m, but experimental evidence has resulted in the generally accepted, empirical value of 1.12. As in Eq. 5 above, for any one instrument

$$p_k = k_3\rho/t^2 \quad \ldots\ldots\ldots\ldots (7)$$

If p_k is subtracted from P in Eq. 5

$$\eta = K(P - k_3\rho/t^2)t = KPt - K'\rho/t \ldots (8)$$

With viscometers in which the driving force is gravity, and therefore dependent on the density of the liquid

$$\eta = C\rho t - K'\rho/t$$

or

$$\eta/\rho = Ct - K'/t$$

or

$$\nu = C(t - C'/t) \quad \ldots\ldots\ldots (9)$$

wherein ν is the kinematic viscosity in stokes and C'/t can be considered a correction to the time of flow through the capillary.

The Bingham Absolute Viscometer (**7**). *(10^{-2} to 10^2 poises):*

This instrument, Fig. 3, is composed of a horizontal capillary connecting two reservoir bulbs located in two vertical arms. Air pressure may be applied to either arm to drive liquid from one bulb to the other through the capillary.

Some of the difficulties observed in practice with capillary viscometers include the effects of drainage, surface tension, and changing hydrostatic head within the liquid reservoir bulbs ahead and behind the capillary portion of the instrument. The Bingham instrument tends to cancel out these effects by having the supply and receiver bulbs the same shape, vertically symmetrical, and located at the same level above the horizontal capillary connecting them. If such construction was exact, the changing head would be balanced by applying pressure greater than represented by the hydrostatic head and terminating the run with head as great as in the beginning but in the opposite direction. This effect is further offset by making measurements in both directions. The design, if ideal, would likewise make the drainage effect constant during the entire runs in both directions. Furthermore, the perimeter of both liquid surfaces would be equal at all times, which would cancel the surface tension effect. End effects are minimized by the magnitude of the length to diameter ratio of the capillary (>300). Accurate filling to constant volume as indicated by the mark below the receiving bulb is facilitated by the spill-over tube above the supply bulb.

For over 25 yr this instrument has been considered a standard for the determination of the absolute viscosity of relatively fluid substances. It is not necessary to calibrate it since its behavior can be interpreted by the use of Eq. 8 where K and K' are dependent upon the dimensions of the viscometer.

The Ostwald Viscometer (10^{-2} *to* 10^{2} *poises*):

This instrument, and its modifications, Fig. 4, is probably among the most popular because of the simplicity of its operation. It is composed of a reservoir bulb located above a vertical capillary whose lower end is connected through a U-tube to the receiving bulb at a lower level than the reservoir. It is usually operated by drawing liquid up into the reservoir bulb and recording the time required for it to flow through the capillary back to the receiver. The standard practice with this instrument (ASTM Method D 445 - 37 T) permits calculation of viscosity from

$$\eta = K\rho t \quad \text{.............} (10)$$

wherein K is a constant obtained by calibration with a liquid of known viscosity. The kinetic energy effect is tacitly neglected for velocities less than 280 cm per min through a 1-mm capillary, yet the same ASTM specification requires consideration of kinetic energy in another capillary viscometer (Ubbelohde) for velocities as low as 40 cm per min through a capillary of the same size. For precision of the order of X per cent, Eq. 9 should be used when C'/t is greater than X per cent of the efflux time. Constants C and C' require calibration with two liquids of known viscosity instead of only one to obtain K in Eq. 10. The standard use of this instrument also neglects surface tension effects which may be appreciable due to the difference in shapes of the supply and receiver bulbs.

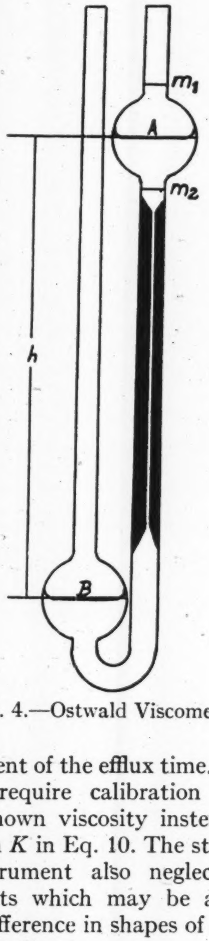

FIG. 4.—Ostwald Viscometer.

Ubbelohde Suspended Level Viscometer (10^{-2} *to* 10^{2} *poises*):

The effect of surface tension on the flow of liquids through tubes is a factor that has been a source of annoyance to the rheologist for at least 75 yr and probably (though unrealized) for the 110 since Poiseuille's first experiments. Various ways of estimating and measuring its effect have appeared, but the general practice has been to avoid it or by operating under pressures high in proportion to it. Attempts to overcome the effect by viscometer design have been made by Sprung, Slotte, Bingham, and others, but one of the more imaginative ones employing a new principle in a relatively simple instrument is that of Ubbelohde (**8**). His instrument, Fig. 5, is of the vertical capillary type, but in operation the liquid is discharged over an inverted surface instead of into a receiver containing excess liquid. The downward traction of the film flowing over this surface is supposed to overcome and balance the upward pull of the surface in the higher reservoir bulb. This is certainly partially true but, due to the changing perimeter of the surface in the upper bulb, it may not be exactly counter at all times. However, since the downward tractive force is influenced by flow velocity, it may overbalance the surface tension at the beginning and underbalance it at the end of a run, producing an overall net balance. Whatever the effect, Ubbelohde's calculations and experiments indicate that, at least within experimental error, the desired result is produced.

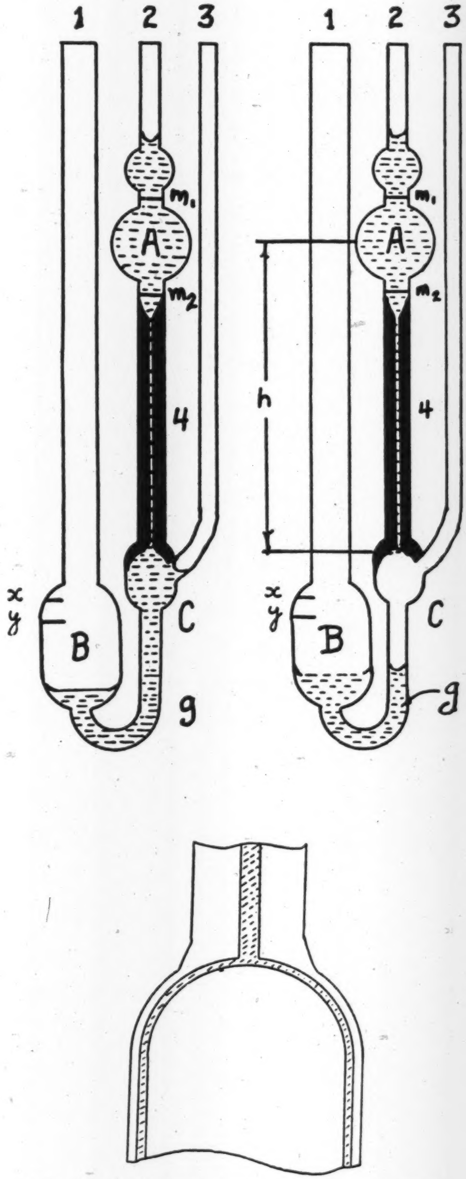

FIG. 5.—Ubbelohde Suspended Level Viscometer.

The apparatus is operated by drawing liquid into the upper bulb while the side tube is closed with the finger, opening the side tube and so forming the "suspended level" at the bottom of the capillary, then allowing flow to begin through the capillary from the reservoir above it.

Ubbelohde points out the necessity of the kinetic energy correction and recommends an equation of the type previously given (Eq. 8). The instrument has been used successfully in the oil and cellulose products industries among others

$$\eta = KPt - K'\rho/t \ldots\ldots\ldots\ldots (8)$$

Triple Capillary Viscometer (10^{-1} *to* 10^{2} *poises*):

This modified Ostwald kinematic viscometer was developed by Anderson, Wright, and Griffin (**9**) for measuring the kinematic viscosities of a wide range of liquid asphaltic road materials, and reduces the necessity for exercise of the operator's judgment in selecting the proper capillary size or pressure required for a given liquid as is often necessary with single capillary viscometers. Adjustment of the liquid volume at the testing temperature enables temperature-independent viscometer constants to be used. This viscometer is readily cleaned. It is especially useful for dark or opaque liquids. However, it is somewhat difficult to fill, and the upper level of the liquid must be precisely adjusted to avoid appreciable loading errors. It is more fragile than the Cannon-Fenske types. It must be calibrated with liquids of known kinematic viscosity.

The essential feature of this viscometer is the use of three capillaries of decreasing diameter connected in series with intervening bulbs (see Fig. 6). The instrument is filled between marks *M* and *M'* with the liquid under test at the testing temperature. The liquid is allowed to rise and the time required between marks *A* and *A'* is recorded. The capillary and bulb dimensions are so chosen that for liquids of 1500 to 15,000 centistokes the efflux time for the first bulb is 50 to 500 sec. If the efflux time for the first bulb is less than 50 sec (or some predetermined minimum time) the liquid is allowed to continue to flow upward through the second bulb, which gives efflux times of 50 to 500 sec for liquids of viscosity range 150 to 1500 centistokes. Similarly the third bulb gives efflux times of 50 to 500 sec for liquids of viscosity range 15 to 150 centistokes. Thus liquids of a thousandfold range in viscosities (15 to 15,000 centistokes) can be measured with efflux times of 50 to 500 sec.

A special filling tube is used for filling the viscometer. This fits into the receiver arm of the viscometer. The liquid is warmed, if necessary, to a temperature sufficient to permit pouring into the filling tube. The liquid will pass through a sieve into the receiver arm and catch at the juncture, *P*, of the narrow tubing to the receiver (see Fig. 6). The flow down the narrow tube is regulated by means of pressure changes caused by the roller clamp which squeezes the rubber tubing connected to the capillary arm. To control the rate of flow so that the upper surface of the liquid approaches *M* about the same time the lower surface approaches *M'*, the ground cap with stopcock is placed on the filling tube.

The lower level of the liquid is left slightly below *M'* while the upper mark is left slightly above *M*. The viscometer is allowed to reach bath temperature and the lower level is brought exactly to *M'* with the roller clamp. The filling tube is replaced by a leveling tube and the upper level brought to *M* by sucking out excess liquid through the fine tip of the leveling tube. The leveling tube is removed and the viscosity determination is started by opening the two-way stopcock on the capillary arm to the atmosphere which allows the liquid to flow up into the first bulb.

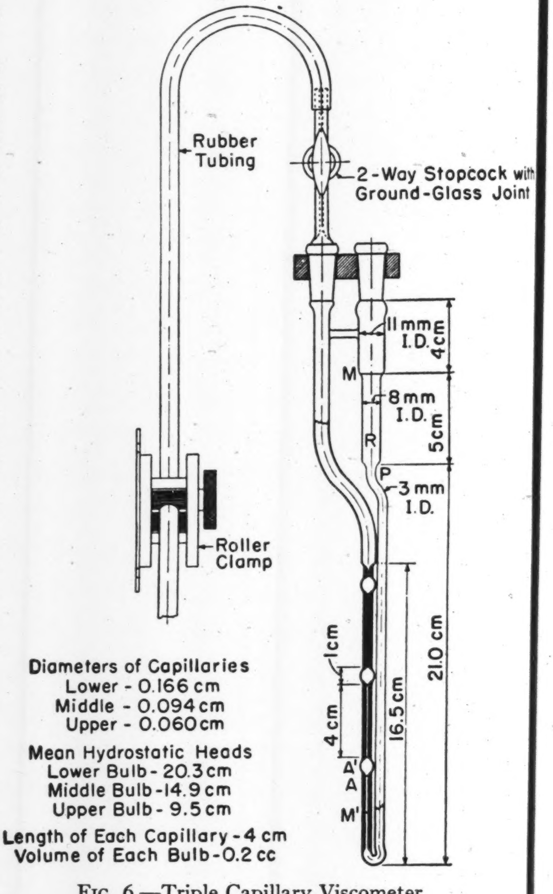

FIG. 6.—Triple Capillary Viscometer.

Kinematic viscosity is calculated from the equation

$$v = Ct \quad \text{..............} \quad (11)$$

where:

ν = kinematic viscosity, centistokes,
C = viscometer constant, centistokes per sec, and
t = efflux time, sec.

(See Eqs. 9 and 10.)

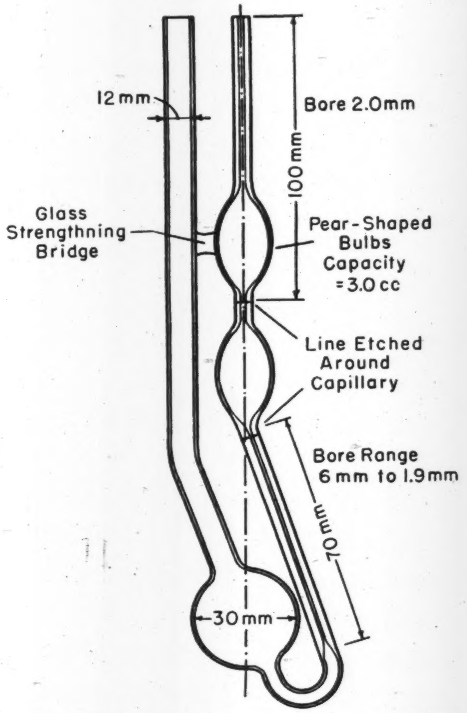
12mm
Bore 2.0mm
100mm
Glass
Strengthning
Bridge
Pear-Shaped
Bulbs
Capacity
=3.0cc
Line Etched
Around
Capillary
Bore Range
6mm to 1.9mm
70mm
30mm

FIG. 7.—Cannon-Fenske Viscometer for Clear Liquids.

Each bulb has a characteristic viscometer constant which differs from the next by a factor of approximately 10. The viscometer constants are determined by use of liquids of known kinematic viscosity. A single standard liquid can be used to calibrate all three bulbs by choosing several different temperatures to give the appropriate viscosity range.

Corrections for kinetic energy, misalignment from verticality, and drainage are negligible for this viscometer (see Ostwald viscometer above). Surface tension effects are minimized to less than 0.1 per cent by making the bulb diameters approximately equal to the inside diameter of the receiver tube. Since the volume of liquid placed in the viscometer is held constant by adjusting at the testing temperature, the viscometer constants are independent of temperature. Loading errors are reduced by use of the leveling tube described, to less than 0.3 per cent for the upper bulb and are lower for the other bulbs.

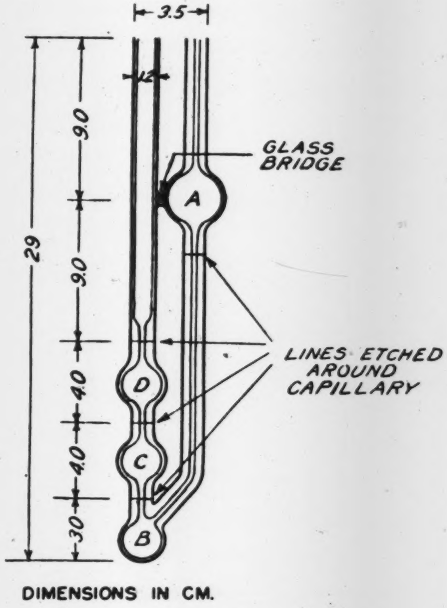
3.5
1.2
29
9.0
9.0
4.0
4.0
3.0
GLASS
BRIDGE
A
D
C
B
LINES ETCHED
AROUND
CAPILLARY
DIMENSIONS IN CM.

FIG. 8.—Cannon-Fenske Viscometer for Opaque Liquids.

Cannon-Fenske Modified Ostwald Kinematic Viscometers (10^{-2} to 10^{2} poises):

Several simple modifications of the Ostwald kinematic viscometer and their use have been described in considerable detail by Cannon and Fenske (**10, 11, 12, 13**) and by ASTM (**14**). These viscometers are widely used in the chemical and petroleum industries. Materials having viscosities from 0.8 to 5600 centistokes can be tested, using a series of six viscometers of increasing capillary diameter.

The viscometers are sturdy, easily cleaned, simple to use, and inexpensive. Opaque liquids can be tested in the type shown in Fig. 8. Corrections for filling errors, kinetic energy, drainage, small misalignments from the vertical, and small surface tension differences are minimized by design of the viscometers. The rapid rate of reaching bath temperature due to small sample size appreciably reduces the time required per determination.

The range of viscosities which can be covered in a single viscometer with reasonable efflux times is 4 to 7-fold, requiring the availability of a series of calibrated viscometers if a wide variety of liquids are to be examined. This also requires prior knowledge of the viscosity range in which the liquid to be tested falls or judgment on the part of the operator in selecting the particular viscometer to be used. The viscometer constant varies with temperature.

Knowledge of density as a function of temperature for liquids under test is required to calculate absolute (in place of kinematic) viscosities. Some uncertainty is introduced if the liquid under test at different temperatures differs appreciably in coefficient of expansion from the calibrating liquid, necessitating a correction which can be treated as a loading error (**12**). It must be calibrated with liquids of known kinematic viscosity.

The more common type of Cannon-Fenske viscometer (**12, 14**), shown diagrammatically in Fig. 7, is used with light-colored or transparent liquids. It consists essentially of two pear-shaped bulbs connected by a capillary tube to a reservoir. The viscometer is so constructed that the vertical axes of the two bulbs and the reservoir coincide to decrease alignment errors.

This viscometer is filled at room temperature by holding it in an inverted vertical position with the capillary side submerged in the liquid under test. Suction is then applied to the other side and both bulbs are filled. The liquid is brought into the working capillary to the etched mark. The viscometer is revolved to its normal vertical position and placed in a constant temperature bath maintained at the testing temperature. When bath temperature is reached, the liquid (which has drained into the reservoir) is drawn up into the upper bulb by suction applied to the capillary side, and the efflux time required for the liquid meniscus to pass from the mark between the bulbs to the mark below the lower bulb on the capillary is measured.

The viscometer shown diagrammatically in Fig. 8 is particularly useful for opaque liquids (**13**) but has been used for lightly colored or transparent liquids also. Since it is designed for opaque liquids, the efflux bulbs are on the bottom. When testing, the liquid flows from reservoir *A* into the pear-shaped 3-ml efflux bulbs on the bottom; hence the meniscus is always in contact with clean unwetted glass and can be seen readily.

This viscometer is filled as described above: the main reservoir, *A*, is filled and the liquid brought into the capillary to the etched line just below *A*. Excess liquid is wiped off the end of the capillary arm and the instrument is inclined slightly to allow the liquid to flow by gravity from the upper capillary into *A*. A corresponding small quantity of liquid is thus discharged into the working capillary leading to bulb *B*. A short piece of rubber tubing closed on one end by a pinch clamp is slipped over the open end of the upper capillary, the viscometer returned to its normal vertical position, and placed in a constant temperature bath. When bath temperature is reached, the pinch clamp is released and the liquid flows by gravity from *A*, through *B*, *C*, and *D*. The times of efflux from the marks above and below bulb *C* and bulb *D* are measured to obtain duplicate measurements.

Kinematic viscosity is calculated from the equation

$$\nu = Ct \qquad (11)$$

where:

ν = kinematic viscosity, centistokes,
C = viscometer constant, centistokes per sec, and
t = time of efflux, sec.

(See Eqs. 9 and 10.)

The viscometer constant is obtained by determining the efflux time for liquids of known kinematic viscosity and substitution in Eq. 11.

Corrections for loading errors, Couette effect, drainage, and small misalignments from verticality are negligible for these viscometers (**12**). Large differences in surface tension between calibrating liquids and liquids being tested necessitate a small correction in effective driving head, but this correction is less than 0.1 per cent if the liquids differ by less than 5 dynes per cm in surface tension. Kinetic energy corrections are negligible for liquids above 2 centistokes if the capillary radius is selected to give suitably long flow times (**12, 14**) (see Ostwald viscometer above). The viscometer constant varies slightly with temperature because of the change in specific volume with temperature when the viscometer is loaded at a fixed temperature. This change in viscometer constant can be calculated from the equation:

$$C_{T_2} = C_{T_1}\left[1 - \frac{V_2 - V_1}{0.785Hd^2}\right]\ldots.(12)$$

where:

C_{T_2} = viscometer constant at temperature T_2,

C_{T_1} = viscometer constant at temperature T_1,

V_2 = total volume of liquid in viscometer at T_2,

V_1 = total volume of liquid in viscometer at T_1,

H = driving liquid head, approximately given by the distance between the centers of the efflux bulb and the reservoir, and

d = working diameter of reservoir.

The above correction amounts to about 0.5 per cent for a 60 C change in temperature for lubricating oils and gasolines.

Bingham and Murray Horizontal Capillary Viscometer (10^2 to 10^4 poises):

Although the Bingham Plastometer (p. 19) is free of drainage error, this problem has been approached in still another way by measuring the rate of filling of an initially empty tube. This interesting converse of the preceding methods was apparently studied first by Decharme (**15**). One of the simpler adaptations of the method that has found considerable application in industry is that of Bingham and Murray (**16**), Fig. 9. It has been employed for the study of such materials as tooth paste (**17**), paints (**18**), pitch (**19**), and nitrocellulose (**20**), as well as oils and greases (**21**). However, this means of overcoming the drainage difficulty enhances that produced by surface tension which tends to aid the advance of the liquid meniscus along the capillary. Detailed mathematical analyses of this effect, as well as those of hydrostatic head and the viscosity of the air ahead of the liquid meniscus, have been presented by numerous authors (**22**). The equations resulting from these studies are fairly complicated, but a simplification of the one given by Bosanquet (**23**) will apply if the rate of entry of the liquid into a capillary under a relatively high driving power is observed. If a constant pressure drop, P, is maintained through the capillary by application of air pressure that is high in relation to the effective pressures due to hydrostatic head and surface tension, the time required for the meniscus to move from one point, L_0, to another, L, in the capillary is

$$t = k\eta(L_0 - L^2)\ldots\ldots\ldots.(13)$$

If the positions L_0 and L are marked on the tube this reduces to:

$$t = k_1\eta/k_2P\ldots\ldots\ldots\ldots(14)$$

wherein $k_1(1/t)$ is the rate of shear, k_2P is the shearing stress, and k_1 and k_2 are constants dependent upon dimensions of the capillary, and are most easily obtained by calibration with a material of known viscosity.

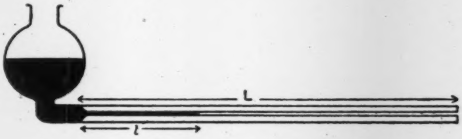

FIG. 9.—Bingham and Murray Horizontal Capillary Viscometer.

The apparatus is usually used at low rates of shear; at higher rates a kinetic energy correction should be applied. A plot of $1/t$ *versus* P for a liquid of *known* Newtonian behavior will indicate the value of

ρ/t below which this is necessary. It will be the value of $1/t$, multiplied by the density of the liquid, below which the plot is curved.

It may be difficult to operate this viscometer at constant pressure drop P without complex auxiliary apparatus. The instrument is equally useful, even more so when studying anomalous flow, if a constant pressure P_0, high in proportion to head and surface tension, is applied to the upstream liquid surface. Under this condition of operation, the shearing stress is constantly changing, the pressure gradient at any time being proportional to P/L where L is the distance of the advancing meniscus from the reservoir end of the capillary. If the capillary is marked at various points L, L_1, L_2, etc., a study of the liquid may be made under changing rates of shear by noting the time the meniscus passes each mark. Bingham and Murray plotted the mean rate of flow $\pi R^2(L_1 - L_0)/t$, where t is the time required for the meniscus to travel between L_0 and L_1, against the log mean average shearing stress $\frac{1}{2} PR[\log (L_1/L_0)]/(L_1 - L_0)$. From such plots they obtained "mobilities" and "yield values."

A simplified manner of obtaining the same information from a calibrated instrument would be to plot L/t, the velocity of flow (of which the shearing rate is a function), against P/L which is proportional to the shearing stress. This is merely representative of a series of analogs of Eq. 14 and is simply a means of obtaining a number of values of P and t from one experiment. Intercepts on such plots would be indicative of yield values, while curvature would be representative of deviation from linear flow. Such a plot would represent

$$\eta = \frac{k_1(P_0 - P)/L}{k_2L/t} \quad \ldots\ldots\ldots(15)$$

wherein k_1 and k_2 are instrument constants and k_1P/L is the yield value (intercept on P/L axis). The constants k_1 and k_2, or simply one constant K, could be obtained from a known Newtonian liquid since they would be valid for both normal and anomalous fluids. Again, kinetic energy must be borne in mind and at high $1/t$ values the relation will become

$$\eta = K(P_0 - P)t/L^2 - K_1\rho/t \quad \ldots\ldots(16)$$

Koppers Capillary Rise Viscometer (10 to 10^5 poises):

Capillary rise viscometers have been in use for many years for determining the viscosities of liquids of high viscosity. Pochettino (**24**) used this method in 1914 to determine the viscosities of bituminous materials, while Flory (**25**) employed it to determine the viscosities of molten polyesters. Rhodes, Volkmann, and Barker (**26**) have extended the mathematical treatment and developed the Koppers Capillary Rise Viscometer as a standardized instrument for the rapid and precise determination of high viscosity in absolute units. Stow, Horowitz, and Elliott (**27**) have further extended the theory and use of this method.

This viscometer is particularly adaptable to high-viscosity liquids and highly colored or opaque liquids. Deviations from purely viscous flow can be indicated and investigated by use of different applied vacua. The instrument is easily filled and cleaned, reaches bath temperature rapidly, requires no calibration, and is available commercially.

The Koppers viscometer is not highly precise for low-viscosity liquids where low vacua must be applied. Some judgment on the part of the operator must be exercised in selecting the range of vacua to be applied in order to give reasonable times of rise. The density of the liquid at the testing temperature must be known.

For more fluid materials and consequent low applied vacua required to give reasonable times of flow, effects due to surface tension, errors in immersed height of the capillary tube, and misalignment from verticality, as well as neglect of the small decrease in level in the sample cup as the liquid level rises in the capillary, become appreciable. Errors in measuring low vacua may introduce large errors. For more viscous materials these effects become small in comparison with the higher vacua applied. Up to about 500 poises a precision of 1 per cent is normally attainable, while for viscosities above this, the precision improves.

The viscometer with accessory equipment is shown schematically in Fig. 10. It consists essentially of a graduated capillary tube whose lower end is immersed in the liquid to be tested. The liquid is contained in the sample cup and a sleeve supports the upper end of the capillary tube. The sample cups, sleeves, and capillary tubes are interchangeable and the entire unit, including bath and vacuum accessories, is available commercially. The capillary tubes are made from precision-bore tubing guaranteed uniform to ±0.01 mm in bore diameter.

In use, the liquid under test is poured into the sample cup, the sleeve and capillary tube connected, and the unit allowed to come to bath temperature. The capillary tube is then lowered until it is immersed to 1 cm in the liquid. Vacuum is applied to the top of the capillary tube through the vacuum reservoir. The time required for the liquid meniscus to rise between two fixed graduations in the capillary tube for the vacuum employed is noted and the vacuum measured on either a water or mercury manometer.

This instrument requires no calibration. Viscosities are calculated from the following equation:

$$\eta = \frac{g r^2 \rho \Delta t}{8\left[\left(\frac{H}{\rho}+\lambda\right) ln \frac{\left(\frac{H}{\rho}+\lambda-l_1\right)}{\left(\frac{H}{\rho}+\lambda-l_2\right)} - (l_2-l_1)\right]} \qquad (17)$$

where:

η = viscosity, poises,

g = gravitational constant, cm per sec per sec,

r = radius of capillary bore, cm,

ρ = density of liquids, g per ml,

Δt = time of capillary rise, sec,

H = vacuum applied, cm H_2O,

λ = depth of immersion of capillary in liquid, cm,

l_1 = initial height of liquid in capillary at start of timed interval, cm, and

l_2 = final height of liquid in capillary at end of timed interval, cm.

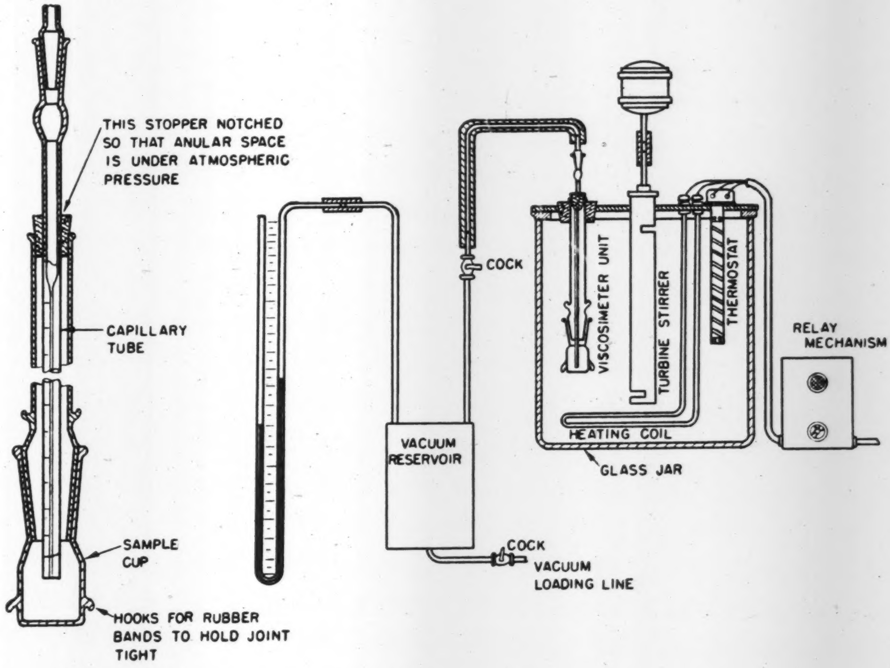
THIS STOPPER NOTCHED SO THAT ANULAR SPACE IS UNDER ATMOSPHERIC PRESSURE
CAPILLARY TUBE
SAMPLE CUP
HOOKS FOR RUBBER BANDS TO HOLD JOINT TIGHT
VACUUM RESERVOIR
COCK
COCK
VACUUM LOADING LINE
VISCOSIMETER UNIT
TURBINE STIRRER
THERMOSTAT
HEATING COIL
GLASS JAR
RELAY MECHANISM

FIG. 10.—Koppers Capillary Rise Viscometer.

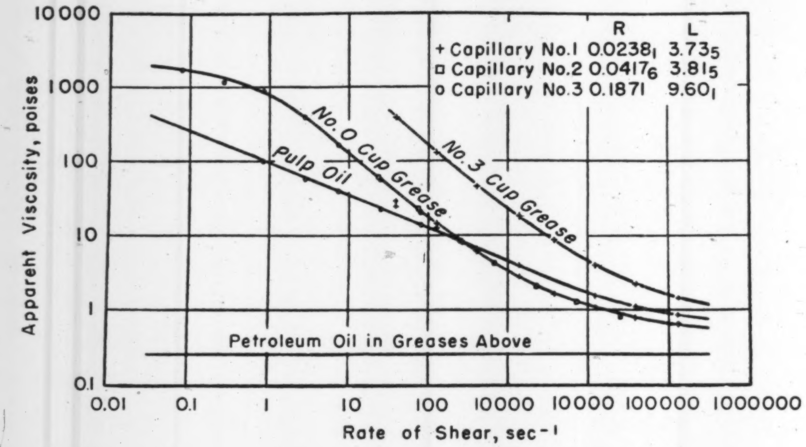

FIG. 11.—Effect of Shear Rate on Grease Viscosities.

If such quantities as r, λ, l_1, and l_2 are kept fixed, the above equation can be simplified and calculations facilitated by use of a graph relating $\frac{\eta}{\rho\Delta t}$ and H for various values of ρ. This is commonly done in routine use of the Koppers viscometer since capillary tubes supplied by the manufacturer are of standardized radius. Charts graphically relating $\frac{\eta}{\rho\Delta t}$ and H for densities ranging from 0.75 to 1.25 are supplied by the manufacturer. If the relative dimensions of the capillary tube and sample cup are changed, correction for change in level in the sample cup may be required (**27**).

Bingham Plastometer (10^2 to 10^{12} poises):

It is at times impractical to use capillary viscometers of the preceding types. Such is the case when studying extremely viscous materials, or substances whose apparent viscosities depend upon rate of flow. Greases, paints, and pitches are examples of such fluids.

Bingham (**28**) recommended a controlled pressure "plastometer" for materials of this nature. It consists fundamentally of a sample cup having a capillary tube outlet through which the material is discharged. Experimental observations with this apparatus consist of measurement of the rate of discharge of the sample by the application of a known pressure. A number of adaptations (**29**) of this principle have given reliable results. Arveson's (**30**) modification is of particular interest for reasons set forth by Merrington (**31**) as shall be shown presently.

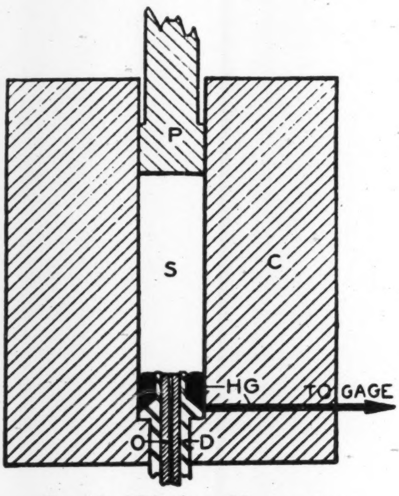

FIG. 12.—Bingham Plastometer.

Interpretation of the data obtained from this type of instrument has been effected by the modified Poiseuille law discussed under capillaries in general

$$\eta = KPt - K'\rho/t \text{..........} (8)$$

However, as pointed out by Merrington,

misunderstanding of the true value of P has led to error in the case of materials having widely different apparent viscosities at different rates of flow (Fig. 11, from Arveson). Since the diameter of the cup is usually much greater than that of the capillary, the pressure drop from fluid surface to capillary entrance has been generally considered negligible. Consequently, the applied pressure has been taken as P, the pressure at the capillary entrance. This may not be even approximately true for materials behaving as mentioned above. The Arveson modification of the plastometer allows measurement of pressure at the capillary entrance as well as at the liquid surface. A schematic diagram of Arveson's apparatus is shown on Fig. 12. Although a constant pressure is maintained by a mechanical system, the application of air pressure should be as satisfactory. The instrument should be used at pressures high in comparison to the changing hydrostatic head in order to facilitate operation at constant rates of flow. The high pressures likewise reduce the surface tension effect to a negligible value.

A further correction to P must be made when the subject material exhibits other than Newtonian flow. This correction can be found by making determinations at several pressures, plotting P *versus* V and extrapolating to zero flow to obtain p, the yield value. The interpretation of the results is then made by

$$Z = K(P - p)t - K'\rho/t \quad \text{(8a)}$$

The viscosity, η, has been replaced by Z, the "consistency," in this equation since it is no longer described by the ratio of total applied force to volume flowing as discussed in the preceding section. Furthermore, Z does not completely describe the flow characteristics of such materials. If the plot of P *versus* V is linear, it is sufficient to report Z and p. If the plot is of higher order (Eq. 3), it is also necessary to report the velocity of flow in the capillary, since $(P - p)/V$ is no longer constant.

An attribute of this instrument that should not be overlooked is its freedom from drainage error since the actual amount of fluid discharged from the capillary is measured.

FALLING BODY VISCOMETERS

The discussions in the preceding section dealt with measurement of the rate of flow of liquids past solids as a means of studying liquid structure and "internal friction" or viscosity. This problem can also be approached by observing the manner in which solids move through liquids. Newton interested himself in this problem as well as in that of fluid flow through tubes. He observed the rates at which fairly large spheres fell through liquids in a tall tank of square section. Analysis of the fall of spheres through cylinders was probably first attempted by Stokes. This work was later expanded by other investigators, among whom Oseen, Ladenburg, and Faxen have contributed much of importance. The motion of solids other than spherical has also been studied. Other investigations have included the motion of plugs half falling and half sliding through close-fitting vertical tubes. The behavior of spheres rolling through inclined tubes is also the basis of a number of interesting studies.

All of these motions appear to be definable by a rather simple formula in which V = velocity of the body, ϕ = fluidity of the liquid $(1/\eta)$, and ρ_S and ρ are solid and liquid densities, respectively,

$$V = A\phi(\rho_S - \rho) \quad \text{(18)}$$

wherein A is a constant dependent on position and dimensions of container and falling body.

Falling Sphere Viscometer (90 to 3000 poises):

This method has long been a standard in industry for measurements on relatively viscous and transparent solutions, especially cellulose products. In fact, the designation of various nitrocelluloses derive from their "falling ball" viscosities, such as 50 sec-, 100 sec-, 400 sec-nitrocellulose, etc.

The method is an extremely simple one and consists in measuring the time required for a glass or, preferably, steel ball to fall a measured distance through the subject fluid contained in a glass tube whose diameter is relatively large compared to that of the ball. The approximate scalar relation between the two is shown on Fig. 13. During the early industrial development of the method, a great deal of attention was paid to the means of releasing the ball exactly at the center of the liquid surface. Experience has shown that balls released by hand near the surface center give check results within 1 per cent for four or five consecutive falls with the balls falling no more than about 3 in. apart. These results have also been found to check those from capillary viscometers to the same precision.

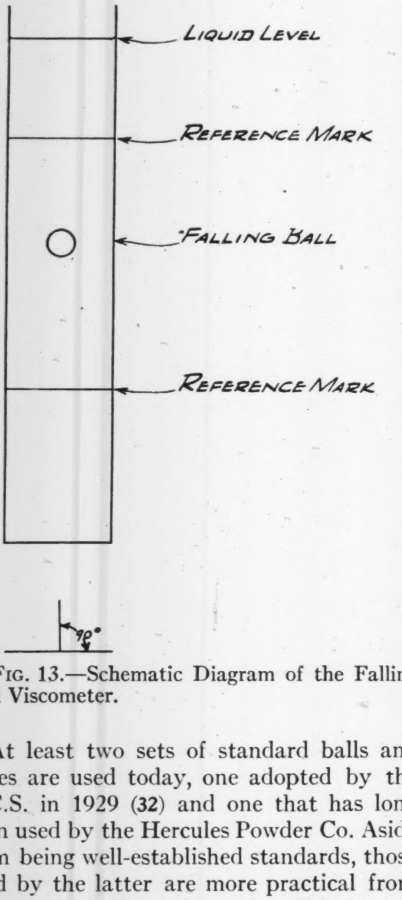

FIG. 13.—Schematic Diagram of the Falling Ball Viscometer.

At least two sets of standard balls and tubes are used today, one adopted by the A.C.S. in 1929 (**32**) and one that has long been used by the Hercules Powder Co. Aside from being well-established standards, those used by the latter are more practical from the point of view of supply. This instrument also may be used for opaque liquids by electrically measuring the drop of metal balls.

The theory applying to the behavior of the falling ball goes back to Newton, although Stokes (**33**) is credited with its primary mathematical treatment. According to Newton a moving body will eventually attain a constant velocity if it is acted upon by a constant force and resisted by a means proportional to the velocity of the body. Stokes expressed the relation between the applied and resisting forces for a sphere falling at low velocities in a liquid of infinite expanse by

$$\tfrac{4}{3}\pi R^3(\rho_S - \rho)g = 6\pi\eta Rv \ldots\ldots (19)$$

wherein R is the sphere radius, ρ_S the sphere density, ρ the liquid density, g the acceleration due to gravity, η the liquid viscosity, and v the sphere velocity. It is usually impractical to use a liquid container so large its dimensions can be considered infinite in proportion to those of the ball. Likewise, it is sometimes desirable to operate at velocities other than "low." Various authors (**34**) have treated upon the effects of higher velocities, tube diameters and ends. A modification of the Faxen (**35**) equation that has been found satisfactory for precision of the order of $\frac{1}{4}$ per cent when velocities of $\frac{5}{16}$-in. steel balls in 1-in. tubes are no more than 1 cm per sec. (viscosities above 90 poises) is

$$\eta = [(2gR^2)/9S](\rho_S - \rho)Ct = \text{viscosity in poises} \ldots (20)$$

where:

$C = 1 - 2.104\,(d/D) + 2.09\,(d/D)^3 - 0.95\,(d/D)^5$,
g = acceleration due to gravity, cm per sec,^{2}
R = ball radius, cm,
S = timed distance of fall, cm,
ρ_S = ball density, g per cu cm,
ρ = liquid density, g per ml,
d = ball diameter, cm,
D = tube diameter, cm, and
t = time of fall through distance S, sec.

For the particular ball and tube sizes given and falling distances of 10 in., Eq 20 reduces to

$$\eta = 0.535(\rho_S - \rho)t \ldots\ldots (20a)$$

If it is so desired, Eq 20 may be written

$$\eta = K(\rho_S - \rho)t \ldots\ldots (21)$$

and K can be determined for any ball and tube combination by calibration with a liquid of known viscosity.

Anomalies of flow can be detected with this instrument by timing balls of various sizes falling in the same tube and by timing the rates of fall in different sections of the tube to detect rate changes.

Hoeppler Rolling Ball Viscometer (10^{-4} to 10^4 poises):

The behavior of a ball rolling inside an inclined tube containing a liquid has received sporadic attention since about 1914.

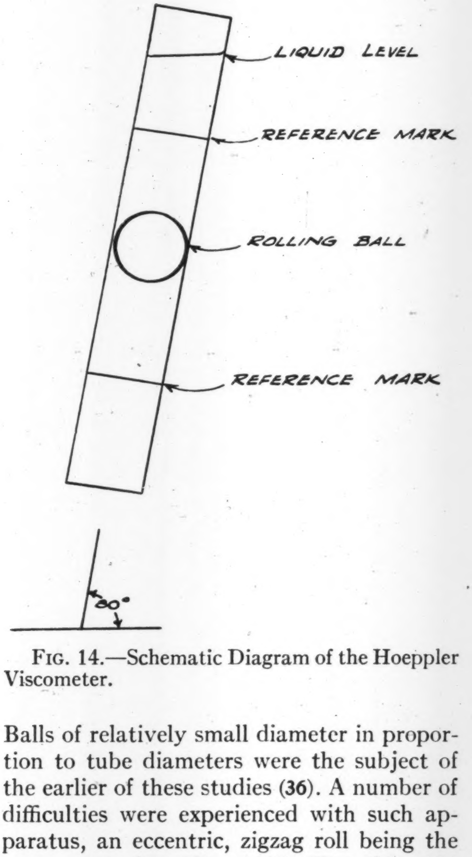

FIG. 14.—Schematic Diagram of the Hoeppler Viscometer.

Balls of relatively small diameter in proportion to tube diameters were the subject of the earlier of these studies (**36**). A number of difficulties were experienced with such apparatus, an eccentric, zigzag roll being the most annoying. In 1933 Hoeppler (**37**) reported a further development of the method to the World Petroleum Congress. His investigations were concerned with the roll of balls whose diameters approached those of the tubes he used. Elimination of the eccentric behavior of the ball was accomplished by this means. He reported the tube to ball ratios (R/r) that gave satisfactory results and remained within practical timing limits. The work covered the ratios from about 1.005 to 12.25 and showed that turbulence resulted over the entire range when ball velocities were greater than 1.7 cm per sec when the tube was inclined at an angle of 80 deg. Turbulence increased with increasing ratio up to $R/r = 1.732$ and then decreased again. At this point, increasing eccentricity of roll began. For the range 10^{-4} to 10^4 poises, the most satisfactory R/r range lay between 1.004 and 1.342 at an optimum angle of 80 deg from horizontal.

Modifications of the instrument (**38, 39, 40**) have been found to be satisfactory for the determination of vapor and liquid viscosities over large pressure and temperature ranges from 0.5 to 1000 psi abs and −30 C. to + 250 C.

By dimensional analysis Hubbard and Brown (**41**) developed an equation relating instrument dimensions, velocity, and viscosity. They showed that

$$R/(D_0^2\rho v^2) \propto D_0 v\rho/\eta \ldots\ldots\ldots\ldots (22)$$

$$R = \text{driving force} = \tfrac{5}{7}\, g \text{ sine } \theta \frac{d^3}{6} (\rho_S - \rho) \quad (23)$$

where:

g = acceleration due to gravity, cm per sec,^{2}
θ = angle of tube from horizontal, deg,
d = ball diameter, cm,
ρ_S = ball density, g per cu cm,
ρ = fluid density, g per ml,
D_0 = "equivalent diameter" = $D - d$,
D = tube diameter, cm,
v = fluid velocity = $Vd^2/(D^2 - d^2)$,
V = ball velocity, cm per sec, and
η = viscosity of fluid, poises.

Expansion of Eq 22.

$$R/D_0^2\rho v^2 \propto D_0 v\rho/\eta$$

$$R/D_0^2\rho v^2 = D_0 v\rho/C\eta$$

gives:

$$\eta = (5\pi/42)C\, g \text{ sine } \theta d(D + d)(\rho_S - \rho)/V$$

where $V = S/t$ (distance of roll/time) or

$$\eta = (5\pi/42)(C/S)\, g \text{ sine } \theta\, d(D + d)(\rho_S - \rho)t \,..\, (24)$$

from which, for any one ball and tube combination,

$$\eta = K(\rho_B - \rho)t \quad \ldots\ldots\ldots\ldots (21)$$

They imply that the coefficient of proportionality, C, is a function of d/D only and should be the same for all ball and tube combinations that have the same R/r value.

Although the works of other investigators (**42**) agree with the proportionality expressed by Eq 22, the constancy of the coefficient seems questionable. Hubbard and Brown in a later work (**39**) themselves report that calibration with a known liquid is the safest practice for obtaining K. The sensitivity of the coefficient, C, to R/r (and possibly to R and r), especially at low values, probably contributes to this discrepancy.

A schematic diagram of Hoeppler's instrument is shown in Fig. 14. The instrument is extremely easy to operate and permits any desired number of check runs simply by turning the whole instrument back and forth on its horizontal axis and timing the roll of the ball between the graduation marks on the tube.

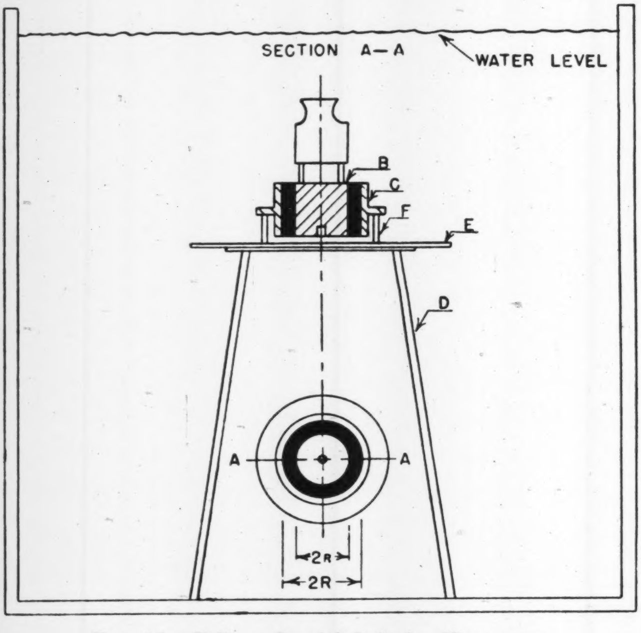

FIG. 15.—Falling Coaxial Cylinder Viscometer.

***Falling Coaxial Cylinder Viscometer* (10^4 *to* 10^8 *poises*):**

This method, first described by Segal (**43**), was used by Pochettino (**24**) for measuring the viscosity of pitch and by Mack (**44**) for determining the viscosity of asphaltic materials. Traxler and Schweyer (**45**) modified the instrument for the rapid evaluation of asphalts.

The falling coaxial cylinder viscometer is an absolute method for essentially simple (Newtonian) liquids only. Where appreciable complex flow or elasticity is exhibited, relative measurements can be made if rigidly standardized conditions are maintained.

The advantages of this viscometer are its low cost and simple construction. Because of its low cost, it is especially adapted to age hardening studies where several viscometers are filled with the material to be tested and stored for various intervals of time. For simple liquids, reproducibility of the method is about ±3 per cent.

The viscometer which is shown diagrammatically in Fig. 15 consists of a solid brass or aluminum inner cylinder, B, and a hollow brass outer cylinder, *C*. The two cylinders are mounted coaxially on an amalgamated brass base plate and the annular space filled with the material to be tested.

To start a determination the viscometer is removed from the base plate and placed so that the flange of the outer cylinder rests upon a supporting brass ring, *F*, which is supported by a brass plate, *E*, and tripod, *D*, and which holds the viscometer in a vertical position in a glass-walled water bath. Under the force of gravity (if necessary, added weights are used) the inner cylinder falls slowly "out" of the hollow cylinder. After a fall of about ¼ cm the viscometer is reversed and the inner cylinder allowed to fall "in" to its original position and "out" another ¼ cm. The rate of movement of the inner cylinder is measured by sighting a cathetometer at a small ring placed on top of the inner cylinder. Several "in-out" readings can be taken on the same sample.

The following equations apply when the material being tested is of essentially the same density as the bath liquid:

$$F = Wg/\pi(R + r)L \quad \ldots\ldots\ldots (25)$$

$$S = 2h/t(R + r) \log_e (R/r) \quad \ldots\ldots (26)$$

$$\eta = F/S \quad \ldots\ldots\ldots\ldots\ldots (27)$$

where:

F = mean shearing stress, dyne per sq cm,
S = mean rate of shear per sec,
η = viscosity, poises,
W = effective weight in grams of applied load (actual weight minus buoyant effect of liquid in bath),
g = gravitational constant,
R = radius of outer cylinder, cm,
r = radius of inner cylinder, cm,
L = length of viscometer, cm,
h = distance of movement, cm, and
t = time for movement h, sec.

For $R = 1.905$ cm, $r = 1.270$ cm, $L = 2.540$ cm, and $g = 980$, Eq. 27 becomes

$$\eta = 24.9\ Wt/h \quad \ldots\ldots\ldots\ldots (28)$$

ROTATIONAL VISCOMETERS

One method of viscometry for which a mathematical treatment was early developed is the rotational viscometer. The material to be tested is contained between two vertical coaxial cylinders. There are two general types of rotational viscometers: (1) one cylinder is rotated at a definite velocity and the torque required to prevent the other cylinder from rotating is measured, and (2) one cylinder is held stationary and the velocity of rotation of the other cylinder under a fixed torque is measured.

If the radii of the cylinders are a and b, the length, L, and a torque, T, is produced on one cylinder by a constant angular velocity, V; on the other cylinder, the viscosity, η, may be calculated by the equation:

$$\eta = T(1/a^2 - 1/b^2)/4\pi LV \quad \ldots\ldots (29)$$

With rotational viscometers of practical length it is necessary to make some kind of end correction. Couette (**46**), who first used the rotational viscometer, and Humphrey and Hatschek (**47**) protected the ends of their inner cylinders with guard rings. Gurney (**48**) and Searle (**49**) calculated the necessary end correction by using two different inner cylinders of different lengths. For materials of high viscosity, Mooney and Ewart (**50**) used a viscometer with the lower ends conical in shape and of such an angle that the mean rate of shear in the end was the same as that in the cylindrical section of the sample.

Garrison Viscometer (10^{-2} to 10 poises):

This instrument has been considerably modified and improved since the original description (**51**). A variety of strain rates were originally obtained by adjustment of the rotor speeds which were measured stroboscopically. Five fixed speeds are now obtained by means of a constant speed motor and a gear change box. Specifications of the latest modification of the instrument have not been published. The range is variable, depending on the cups and rotors used as well as the speeds employed. In general, it is from one to 200 centipoises but may be extended by choice of cup and rotor combinations.

The Garrison Viscometer is primarily advantageous for the evaluation of thixotropic fluids. The wide range of shear rates (9:1) available is useful for this type of measurement. Also, the constant speed of rotation of the rotor can be maintained for any time desired and measurements made at any intervals without altering the strain rates. The speeds can be changed in less than one second. Measurements are not subject to errors caused by bearing friction, since the cup is suspension-mounted; oscillations are eliminated by oil damping. The instrument can be set and read to about 0.1 per cent for viscosities of the order of 100 centipoises.

The viscometer was designed primarily for the evaluation of non-Newtonian fluids such as clay suspensions. It, therefore, is not provided with precise temperature control. However, water, from a thermostatically controlled bath, may be circulated about the cup while the measurement is in progress. In the case of non-Newtonian fluids the precision obtained is limited by the effect of time of shearing on the sample.

The material being tested is held in a stainless steel cup and is subjected to shear by a rotating inner member. Speeds of rotation are fixed and are easily selected by turning a dial on a compact gear box. Speeds available are: 900, 600, 400, 225, and 100 rpm. The cup is suspension-mounted, which eliminates friction corrections; the torque is read from a vernier on the head of the suspension wire. This viscometer is calibrated with known liquids at all speeds and the viscosities are read from a calibration chart.

Brookfield Synchro-Lectric Viscometer (10^{-2} to 10^3 poises):

The design of this viscometer is not described in the literature but it is commercially available. Its use with asphalt emulsions has been described (**52**). The Brookfield viscometer is especially adapted for use as a portable machine, and when it is desired to evaluate complex liquids at different rates of shear.

The Brookfield Model LV Synchro-Lectric viscometer measures viscosity by the drag produced upon a spindle rotating at a definite constant speed while immersed in the liquid under test and indicates viscosity on a rotating dial by a pointer which is attached to the spindle shaft. The reading obtained represents the tension produced in a spiral spring, the core of which is directly connected to the spindle shaft while the outside terminal is connected to the dial. In turn, the dial is directly connected to the constant speed motor.

Four spindles of different sizes are provided. By means of a leaf spring gear shift, four speeds are obtainable. Hence, four different rates of shear (proportional to 6, 12, 30, and 60 rpm) are obtainable with each spindle. This viscometer is calibrated for immersion in an infinite body, but is equipped with a guard. Viscosity is equal to the dial reading times a constant which depends upon the spindle and angular velocity used.

Stormer Viscometer (1 to 10^3 poises):

The Stormer was one of the first technical rotational viscometers (**53**). It is useful where simplicity of operation and ease of cleaning are primary requisites.

In this viscometer the velocity of a rotating cylinder, actuated by a weight, is measured by a revolution counter attached to the spindle. Various types of rotors such as cylinders, fork paddles, and propeller paddles are used, but absolute viscosity values can be obtained satisfactorily only when a cylindrical rotor is used.

Viscosity in absolute units can be obtained from a plot of time in seconds for 100 rpm against absolute viscosity determined for a series of standard liquids.

Precision-Interchemical Rotational Viscometer (10 to 10^3 poises):

This viscometer and its use were described by Green (**54, 55**). The instrument can be used with materials having viscosities from 1 to 2500 poises and yield values from 1 000 to 180,000 dynes per sq cm.

This viscometer is especially adapted to the evaluation of thixotropy, plastic viscosity, and yield value of materials such as printing inks, paints, and varnishes, but it is a delicate and complicated instrument. The viscosities of standard oils can be checked within ±1 per cent. Consistencies of thixotropic inks can be duplicated within ±5 per cent using a standard procedure.

The cup of this viscometer is rotated at various angular velocities between 10 and 400 rpm and the bob is stationary, being suspended from a helical spring. The viscometer is designed to minimize the effects of plug flow and turbulence. The end effect caused by the bottom of the bob is about 2 per cent of the entire shearing effect. The instrument is provided with a bath which maintains a temperature of ±0.2 C.

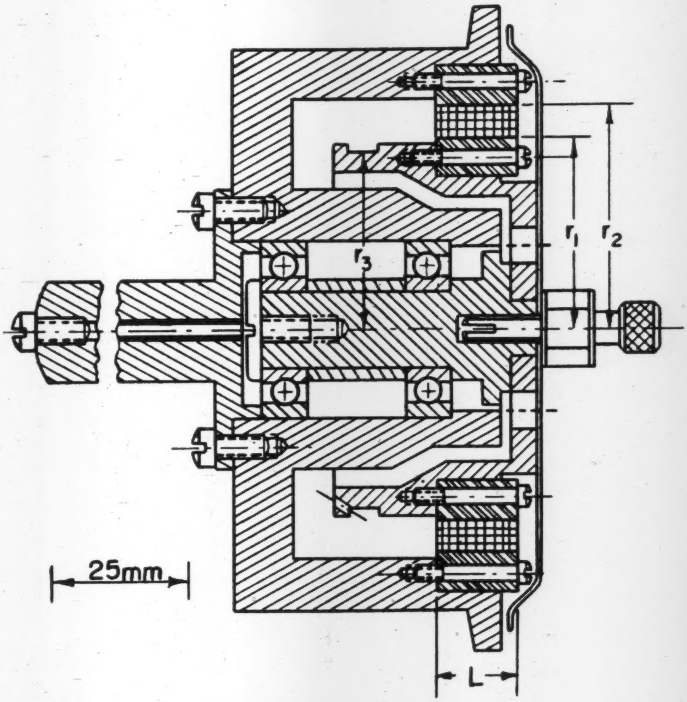

FIG. 16.—Open End Rotating Viscometer.

In studying thixotropy by the "hysteresis loop" method, a series of rapid measurements are made at increasing and then decreasing rates of shear.

Plastic viscosity, U, (reciprocal of mobility) and yield value, f, are calculated from the down curve on the shearing stress (torque) *versus* rate of shear (angular velocity) plot by the equations:

$$U = (T - T_2)S/W \quad \text{.......}(30)$$

$$f = CT_2 \quad \text{................}(31)$$

where:

T = torque,

T_2 = intercept on torque axis,

W = angular velocity, and

S and C are instrumental constants depending upon the dimensions of the viscometer.

The coefficient of thixotropic breakdown, M, can be obtained from the area of the "hysteresis loop" or from two down-curves with different top velocities (**55**).

MacMichael Viscometer (10^2 to 10^5 poises):

The MacMichael viscometer was one of the early technical instruments (**56**) and is still used widely in various industries.

Viscosity is obtained from the angle that a bob, suspended in the liquid by a standard wire, is rotated when the container is turned at a definite velocity. The viscometer is supplied with a disk plunger and a large cup for materials of low viscosity, and different sizes of cylindrical plungers with a small cup for heavy liquids. Four wires are supplied and speeds from 9 to 40 rpm are obtainable by means of an adjustable speed motor. A modification of the MacMichael viscometer is specified by ASTM. Designation D 115 for determining the viscosities of insulating varnishes.

The viscometer is calibrated with standard oils, and close temperature control is not possible with the unmodified MacMichael viscometer.

Open End Rotating Viscometer (10^4 to 10^8 poises):

The design and use of this simple concentric cylinder instrument are described by Ford and Arabian (**57**). The apparatus is especially adapted to the evaluation of asphalts and similar materials exhibiting high viscosity, complex flow, and elasticity. No end corrections are necessary, which simplifies the mathematical treatment. Recovery can be measured. However, it should be pointed out that the annular space is difficult to fill with very viscous materials having large coefficients of expansion, since such materials would usually have to be heated to be poured into the annular space and may shrink away from the rings on cooling to bath temperature. The material to be tested must adhere to the rings to avoid slippage and must not be affected by the liquid used in the constant-temperature bath since they are in direct contact. The rings must be properly aligned when filled to allow them to be mounted on the apparatus without having to twist to obtain alignment. Light oil which fills the bearing housing can become contaminated with the bath liquid.

The open-end viscometer, shown in Fig. 16, is an instrument in which an annulus of material being examined is sheared between concentric cylinders. The outer ring is stationary and the inner ring, revolving on ball bearings, is rotated by weights attached to a cord wrapped around the driving pulley which is rigidly fastened to the inner cylinder. The cord passes over an idler pulley fastened to the same rod which supports the viscometer. The viscometer is submerged in a constant-temperature bath. Deflections of the inner cylinder relative to the outer cylinder are read, with the aid of a pointer mounted on the inner cylinder, on the engraved circumference of the outer cylinder. Small deflections can be magnified optically by means of small mirrors mounted on the hexagonal faces of the nut which holds the pointer in place.

The two concentric rings which hold the sample are removable and rings having varied annular spacings can be used. The concentric rings are filled with the material to be tested, with the use of filling blocks to maintain concentricity.

Measurements are made by hanging various weights on the cord and recording angular deformation and time. If elastic rebound is to be measured, the angle of rebound is measured as the driving weight is released. Recovery can be followed by recording the angular recovery and time. The optical magnification system is useful for the latter two measurements.

Viscosity is calculated from the dimensions of the instrument and the speed of rotation for a given load by means of the following equation:

$$\eta = \frac{360\ W\ g\ r_3\ (r_2 - r_1)}{4\pi^2\ r_0^3\ L\ \theta/t} \quad \ldots\ldots(32)$$

where:

η = viscosity, poises,
g = acceleration due to gravity, cm per sec per sec,
W = net driving weight, g,
r_3 = radius of driving pulley, cm,
r_2 = outer radius of annular ring, cm,
r_1 = inner radius of annular ring, cm,
L = depth of annular ring, cm,
θ = angular deflection, deg, and
t = time, sec,

and

$$r_0 = \left(\frac{2r_1^2\ r_2^2}{r_1^2 + r_2^2}\right)^{\frac{1}{2}}$$

The main shearing stress, S_0, is the shearing stress at the radius, r_0 and is given by:

$$S_0 = \frac{W\ g\ r_3}{2\pi r_0^2\ L} \quad \ldots\ldots\ldots\ldots(33)$$

Corrections for friction in the pulleys must be made to the driving weight to obtain the net driving load. Friction corrections for varied loads are obtained by determining

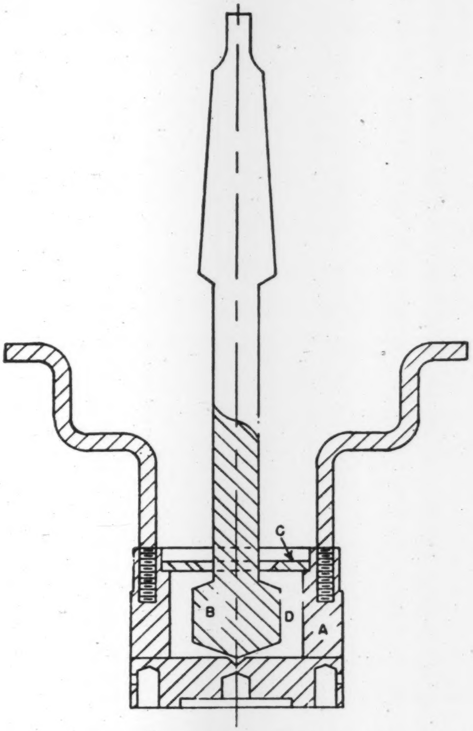

Fig. 17.—High Consistency Rotational Viscometer, Rotor, Stator, and Lid.

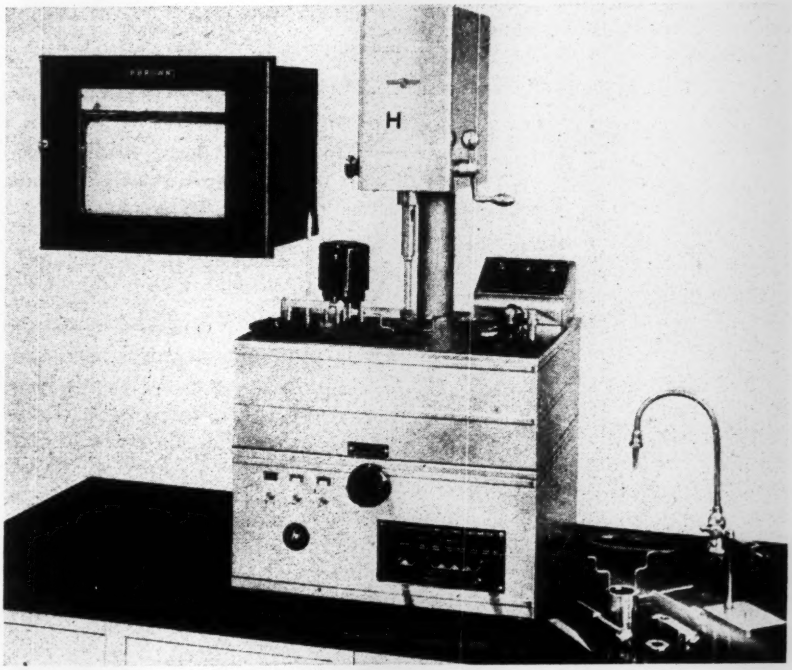

Fig. 18.—High Consistency Rotational Viscometer.

the extra weight required to just start rotation when two equal weights are hung over the pulleys.

Since the mirrors forming part of the optical system for magnification of small deflections are not on the axis of the instrument and a reflected light beam will be refracted entering and leaving the bath, the optical system is best calibrated while the viscometer is submerged in the bath.

High Consistency Rotational Viscometer (10^3 to 10^9 poises):

An earlier form of this rotational viscometer has been described (**58**). The equations used for calculating viscosity are based on those developed by Mooney and Ewart (**59**).

The viscometer is especially adapted to the evaluation of asphalts and similar materials of high viscosity which often exhibit complex flow and elasticity at atmospheric temperatures. Considerable time is required, however, for evaluating materials at the upper limit of the range of this viscometer (over about 10^8 poises). Relaxation one-half times less than 5 sec cannot be measured.

The standard deviation of duplicate viscosity determinations has been found to be about 3 per cent. The degree of complex flow, "*c*," should check within ±0.05 units and is reported to the nearest 0.05. Relaxation one-half times on duplicate samples check within about ±10 per cent except for very low values.

This instrument is illustrated in Figs. 17 and 18. The outer chamber, *A*, is rotated at a constant angular velocity (ten velocities, between 0.0696 and 2200 rpm are available in the machine shown in Fig. 18) and the torque required to prevent the stator, *B*, from turning is measured. Torque is transmitted from the stator to a spindle and is measured by a Statham gage which is enclosed in the head (*H* of Fig. 18). Torque is recorded on a strip chart potentiometer. The viscometer is provided with a bath which maintains a temperature of ±0.05 C.

The top and bottom of the stator are cones of such an angle that the mean rate of shear is essentially the same at the ends as at the cylindrical part of the annulus, *D*. The lid, *C*, helps prevent elastic materials from pulling away from the stator under the shearing action.

Materials having complex flow are measured at two or three rates of shear. A special chart is provided for plotting log shearing stress (torque) *versus* log rate of shear (angular velocity) and the viscosity is read directly from a line on the chart which represents a power input of 1000 ergs reciprocal sec reciprocal cu cm.

A relative measure of elasticity is obtained by studying the relaxation of the stress (torque) after shearing is stopped. Although certain materials do not follow Maxwell's law, an indication of elasticity is obtained by the time it takes the stress to drop to one-half the value at the time that shearing was stopped.

For the particular size viscometer used, viscosity in poises is equal to 4950 times the torque in gram centimeters divided by the angular velocity in revolutions per hour.

The degree of complex flow can be evaluated by the change in viscosity with change in rate of shear. Laminar flow for some complex liquids such as asphalts can be expressed by the equation:

$$M = F/S^c \quad \text{............} (34)$$

where:

F = shearing stress, dyne reciprocal sq cm,
S = rate of shear, reciprocal sec,
c = slope of log S *versus* log F plot, and
M = value of F when $S = 1$.

Equations of this type are discussed by Barr (**60**). If the material is a simple (Newtonian) liquid, the constant "*c*" is unity and *M* is the viscosity in poises. The value of "*c*" is a measure of complex flow and can be obtained readily from the plot of log *F* *versus* log *S* by the use of a special protractor.

VIBRATIONAL VISCOMETERS

During recent years the mechanical behavior of plastics and polymer solutions subjected to small oscillating deformations has been of increasing interest. In general, a polymer solution subjected to sinusoidal shear stress exhibits both rigidity and viscosity, the rigidity and viscosity being frequency dependent. The rigidity is zero at low frequencies but increases with increasing frequency and approaches some definite value at high frequencies. At these high frequencies, the viscosity is small, but it increases with decreasing frequency and approaches the conventional steady flow viscosity at low frequencies (**61**).

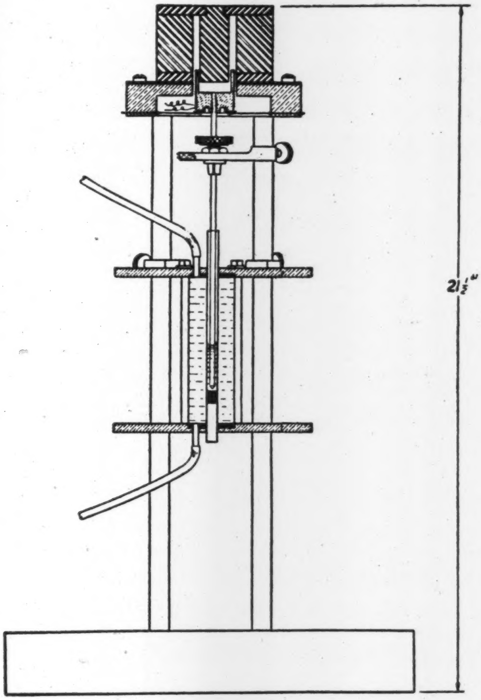

FIG. 19.—Electromechanical Transducer.

Electromechanical Transducer (1 to 10^3 poises):

An electromechanical transducer (energy transfer apparatus) of the type developed by Smith *et al.* (**62**) is suitable for measuring the frequency dependence of shear rigidity and viscosity of polymer solutions at audio frequencies. Low molecular weight, nonthixotropic fluids exhibit no rigidity in the low audio-frequency range, and the measured viscosity equals the steady flow viscosity. Thus, a transducer can be used to determine the viscosities of such fluids whose viscosities are in the range of one to a few hundred poises.

A schematic diagram of a transducer is shown in Fig. 19. A metal rod is driven axially in a sample tube, shearing the solution to be studied across an annular gap of about 1 mm. The top of the rod is fastened to a cylinder of Lucite, which is machined to leave a thin shell on which is wound a coil of wire. The rod and coil are supported in the air gap of a permanent magnet from a loudspeaker by a brass disk attached to four metal rods fastened to a brass ring. It is essential that the moving system be very rigid, although light, and that there be no friction where the coil moves in the magnetic field. The sample is contained in a glass tube surrouned by a water jacket. The tube and jacket can be lowered for filling and emptying; when in use they are clamped with the end of the rod near the bottom of the tube, the depth of immersion being varied by changing the volume of fluid in the tube. The various parts of the apparatus are mounted vertically on a heavy base. The whole mounting must be extremely rigid to prevent anomalous frequency-dependent phenomena from appearing, caused by slight compliances in the mounting.

The coil acts as one arm of an impedance bridge and is driven by current supplied by a signal generator. The frequency range in which measurements can be made is determined by the natural resonance frequency of the rod and the construction of the apparatus. Most accurate measurements are made at frequencies somewhat below, and extending up to, the resonance frequency. The electrical impedance of the coil when in motion is measured and the viscosity of the fluid is calculated from the change in impedance that occurs when the mass of the moving system is varied. A mechanical resistance, R_M, defined as the shearing force in phase with the velocity divided by the velocity, can be calculated from the equation

$$R_M = \frac{(Bl)^2 \times 10^{-9} (R - R_0)}{(R - R_0)^2 + (X - X_0)^2} \quad .(36)$$

where B is the magnetic field strength in gauss, l the length of wire in the coil, R and X the electrical resistance and reactance of the coil when in motion, and R_0 and X_0 the same quantities when the rod is rigidly clamped. As it is difficult to measure R_0 and X_0 directly, the mass of the moving system is varied at constant frequency by clamping various weights onto the rod. The locus of X plotted against R is a circle whose radius is $(Bl)^2 \times 10^{-9}/2R_M$. The viscosity is calculated from R_M using the equation

$$\eta = R_M/a'L \quad \ldots\ldots\ldots\ldots(37)$$

where L is the depth of immersion of the rod, η the viscosity, and a' a dimensionless apparatus constant calculated using the equation:

$$a' = \frac{2\pi\left(\dfrac{r_a^2 + r_i^2}{r_a^2 - r_i^2}\right)}{\dfrac{r_a^2 + r_i^2}{r_a^2 - r_i^2}\,[\log r_a/r_i] - l} \quad \ldots(38)$$

where r_a and r_i are the radii of the sample tube and rod, respectively. In order to eliminate possible end effects, R_M is usually measured at several values of L and the viscosity is calculated from the slope of a plot of R_M against L using the equation:

$$\eta = \frac{1}{a'}\,\frac{dR_M}{dL} \quad \ldots\ldots\ldots\ldots(39)$$

The apparatus constant $(\mathrm{Bl})^2$ can be determined experimentally (**62**) or can be obtained from measurements on a fluid of known viscosity.

Viscosities obtained from transducer measurements agree within 5 per cent or better with values obtained by conventional methods.

Torsional Crystal Viscometer (10^{-2} to 10^3 poises):

Although most measurements of viscosity have been made by flow methods, it has recently been found possible to measure viscosities of liquids at high frequencies by dynamic methods. One method makes use of a torsional crystal for which all the motion is tangential to the surface. Such a crystal will set up a very highly damped viscous wave in the liquid.

For light liquids such as carbon tetrachloride which has a density of 1.595 and a viscosity at 25 C. of 0.0098 poise, the attenuation is large, so that the shearing stress is appreciable for only a few thousandths of a centimeter from the crystal surface. Although the attenuation for such waves is too high to permit their wave propagation properties to be measured, they do introduce a loading effect on the crystal which can be measured by the increase in the resonant resistance and the decrease of the resonant frequency of the crystal as it is changed from a vacuum to the medium under investigation.

The use of such a crystal for measuring viscosities was discussed in former papers (**63, 64**) and it was there shown that the change in the measured electrical resistance, ΔR_E, and the lowering in frequency Δf, determined the mechanical resistance R_M per sq cm and the mechanical reactance X_M per sq cm for the viscous wave according to the formulae

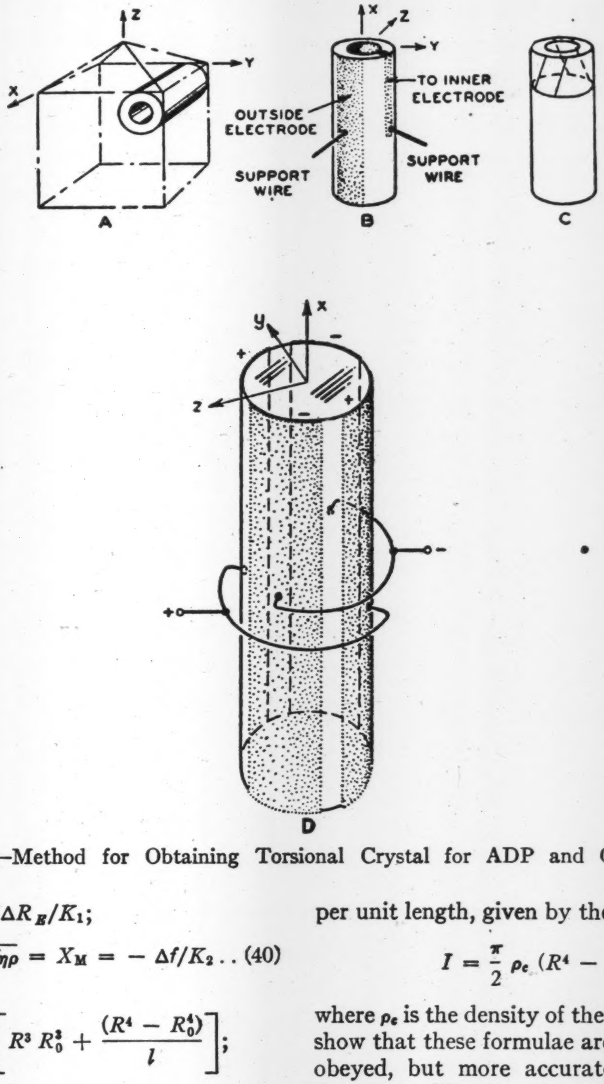

FIG. 20.—Method for Obtaining Torsional Crystal for ADP and Quartz.

$$\sqrt{\pi f \eta \rho} = R_M = \Delta R_E / K_1;$$

$$\sqrt{\pi f \eta \rho} = X_M = -\Delta f / K_2 \quad \ldots (40)$$

where:

$$K_1 = \frac{r}{2\pi f_R^2 C_0 I} \left[R^3 R_0^3 + \frac{(R^4 - R_0^4)}{l} \right];$$

$$K_2 = \frac{R^3 + R_0^3 + (R^4 - R_0^4)/l}{2I}$$

where η is the viscosity of the liquid of density ρ, f is the frequency, r is the ratio of capacitances of the crystal determined by measuring the resonant frequency f_R, the antiresonant frequency f_A, and setting $r = f_R / 2(f_A - f_R)$, C_0 is the static capacitance of the crystal in farads, R the outside radius, and R_0 the inside radius of a torsional crystal, its length and I the moment of inertia per unit length, given by the formula

$$I = \frac{\pi}{2} \rho_c (R^4 - R_0^4) \quad \ldots\ldots (41)$$

where ρ_c is the density of the crystal. Checks show that these formulae are approximately obeyed, but more accurate work can be done by calibrating the crystals in known light liquids.

The method for making a torsional crystal from ammonium dihydrogen phosphate (ADP) is shown by Fig. 20. Here the crystal is cut with its length along the X crystallographic axis, and a hole is bored along this axis. A cylinder is then turned as shown by Fig. 20B. Since the mode of motion generated is a face shear mode when the field is impressed along the Z crystallographic axis, the plating is made continuous on the inside cylindrical surface and is divided into two 90-deg segments on the outside surface which are connected together electrically. The center lines of these two surfaces lie along the *Z*-axis. As shown by Fig. 20C, the field in one segment produces a shear in one direction, while the field in the other segment produces a face shear in the other direction. The result is a torsional motion of the crystal as a whole. As shown by Fig. 20B the crystal is supported by three wires glued to the crystal surface.

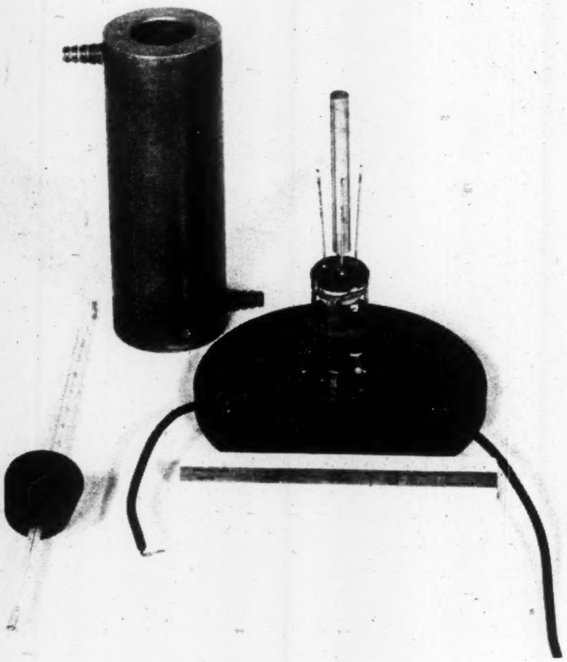

FIG. 21.—Torsional Crystal Viscometer.

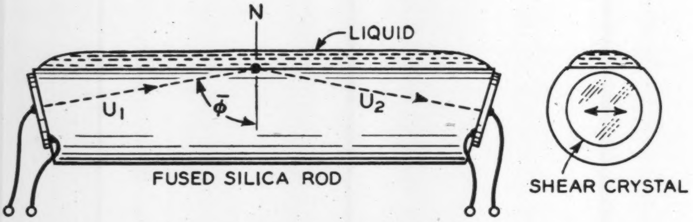

FIG. 22.—Shear Wave Crystal Viscometer.

The *ADP* crystal on account of its high electromechanical coupling is especially adapted to measuring high viscosities. For lower viscosities, i.e., up to a poise, a quartz crystal in torsional motion can also be used, and since this is much more stable with temperature and can be supported by wires soldered to the surface by well-known techniques, this provides a more satisfactory arrangement. As shown by Fig. 20D, by using a cylindrical crystal with its length along the *X*-axis and with four electrodes whose central lines make angles of 45 deg with respect to the *Y*-axis, with opposite pairs connected together, a torsional mode can be generated in the crystal.

A typical container for measuring viscosity is shown by Fig. 21. The crystal mounted in a plug-in base is placed in a sealed container. In the most accurate measurements the crystal is carefully cleaned, after which the chamber is evacuated and the resonant frequency and the resistance at resonance measured. The liquid to be measured is then introduced through stopcocks connected to the chamber, and the new resistance at resonance and the new resonant frequency determined. For a very viscous liquid, point by point curves such as resistance *versus* frequency and capacitance *versus* frequency may be made, and the resistance is taken at the lowest point while the resonant frequency is most accurately determined as the frequency for which the capacity is half way between the two extremes. A faster method which has recently been employed is to put the crystal in a capacity-resistance bridge and balance the capacity at a frequency 50 per cent or more higher than the resonant frequency, which will eliminate the effect of the shunt capacity. Next the paralleling resistance of the bridge is set at a somewhat lower value than the resistance of the crystal resonance and the frequency is changed until the bridge is most nearly balanced. This will be the resonant frequency since at this frequency the reactance will be zero. The resistance is then increased until the bridge is balanced, and the value read off can then be related to the viscosity by means of Eq 40. It should be noted that since the change in frequency between the crystal in a vacuum to the frequency in the liquid is small, it requires a very accurate oscillator if the viscosity is to be measured by a frequency change, while a relatively simple bridge and oscillator can be used if the viscosity is to be measured by the resistance change. Checks with known liquids made at Bell Laboratories and at the Franklin Institute (**65**) have shown that viscosity measurements can be made to better than a per cent if a good temperature control and a good cleaning technique are used.

On account of the extra apparatus required, it appears likely that *the most promising uses for this equipment will be in measuring very small samples of liquid and in continuous recording for the control of manufacturing processes.* On account of the small penetration of shear waves into the surrounding liquids, saturation effects are obtained with liquid layers only a few thousandths of a centimeter thick and hence this method can measure the viscosities of a small amount of a liquid. If a crystal is put into a pipe through which a liquid is flowing, and an automatic bridge balancing system is employed, instantaneous readings can be made and the process controlled if it depends on the viscosity of the liquid.

When long chain polymer liquids having viscosities over 20 poises are measured, it has been found that the liquids have a type of configurational elasticity as well as a shear viscosity. The presence of the shear elasticity is shown by the nonequality of the resistance and reactance terms, and by observing the variation of these quantities over a frequency range, the value of the elasticity can be evaluated. The presence of shear elasticity limits the frequency range that can be used for measuring viscosity alone but gives added information regarding the constitution of the liquids. By this means, the properties of long chain polymers in solution have been studied (**66**), and it has been shown that single molecules not appreciably entangled with other molecules have three relaxation frequency ranges which have been tentatively identified as a configurational relaxation of the molecule as a whole, a relaxation due to interchain entanglement, and finally a relaxation due to the freezing of the chain joints.

The torsional crystal method can be employed in the frequency range from about 10 kc to perhaps 500 kc. Above this frequency the crystals become too small to be realized physically, and another method has been devised (**67**) which is based on the reflection of a shear wave in a fused quartz rod covered at the reflecting face by a layer of the liquid to be measured. In its most sensitive embodiment the arrangement shown by Fig. 22 is employed. Here shear waves having their particle motion parallel to the top reflecting surface are reflected at an angle from the surface, and the shear properties of the liquid are measured by determining the change in amplitude and phase caused by putting a layer of the liquid on the reflecting surface. By this means it has been shown that light liquids have the same viscosity up to 50 mc (within the experimental error of about 5 per cent) that they do when measured by flow methods, while polymer liquids have two relaxation ranges, the first due to a quasi-configurational elasticity of the liquid, while the second range is caused by the appearance of a crystalline elasticity.

PARALLEL PLATE PLASTOMETER (10^4 TO 10^9 POISES)

Conventional instruments, such as ordinary capillary or falling ball viscometers, are not generally applicable for the measurement of very high viscosities. Viscosities of this magnitude are of considerable practical interest since they represent the range encountered for many thermoplastics at their processing temperatures. In order to make the necessary determination, an instrument must be capable of either measuring very low rates of deformation or applying very high loads on the sample. The parallel plate plastometer is of the former type, operating at shear rates in the range .01 to .03 reciprocal sec at pressures up to 250 psi. This instrument is essentially a refinement of the Williams (**68**) plastometer in which the mathematics necessary to reduce the data to fundamental quantities have been worked out (**69, 70**). A method for separating the elastic, delayed elastic, and viscous properties has also been developed (**71**). This latter is of particular interest since many commercial thermoplastics (that is, polyethylene, polystyrene, cellulose acetate) exhibit deformation behavior at elevated temperatures, which is the result of the simultaneous response of these deformation components.

A drawing of the parallel plate plastometer, developed in the Bakelite Corp. Development Laboratories and manufactured by the Tinius Olsen Testing Machine Co., is shown in Fig. 23. The instrument consists of two circular plates, the lower one being fixed and the upper one movable. The upper plate is attached to a vertical shaft which is loaded by means of a weighted beam. The whole instrument is housed in a temperature-controlled oven.

The sample to be tested usually consists of a cylinder about ⅝ in. in diameter and ¼ to ½ in. high, although the size is not critical. It is acceptable to use a sample built up from a number of disks die-cut from a thin sheet. The sample is placed in a *C* clamp between two sheets of .001-in. cellophane (or other suitable material) and put in the oven for preheat. At the end of the preheat the sample is placed between the plates and the desired load is applied. A timer is started simultaneously and the separation of the plates noted, from the dial gage mounted on top of the vertical shaft, as a function of time. It is necessary to correct these readings for the thickness of the cellophane.

The complete development of the equation necessary to reduce the plate separation-time data to fundamental qualities is given in previous papers (**70, 71**). It is shown that, for purely viscous materials, when the material completely fills the space between the plates,

$$\frac{1}{h^2} = \frac{4W}{3\pi\eta a^4}\,t + C_1 \quad\cdots\cdots (42)$$

and when the material does not fill this space,

$$\frac{1}{h^4} = \frac{8\pi W}{3\eta V^2}\,t + C_2 \quad\cdots\cdots (43)$$

where:

h = plate separation, cm,
W = load on sample, dynes,
a = radius of plates, cm,
t = time, sec,
V = sample volume, sq cm,
η = viscosity, poise, and
C_1, C_2 = constants of integration.

Almost invariably conditions are, or can be, most easily adjusted, so that the latter situation, represented by Eq 43 is realized. It is found that C_2, the constant of integration, is the value of $1/h^4$ at time equals zero and can be neglected. The constants of the equation are also conveniently lumped together in the form of a "normalizing factor," *F*, so that,

$$\frac{1}{h^4} = \frac{1}{F} \cdot \frac{1}{\eta}\,t \quad\cdots\cdots\cdots (44)$$

or

$$F\left(\frac{1}{h^4}\right) = \frac{1}{\eta}\,t \quad \ldots\ldots\ldots (45)$$

where:

$$F = \frac{V^2 \times 10^{-6}}{8.21\,W'}$$

and

W' = load in kilograms

Thus the reciprocal of the plate separation taken to the fourth power is plotted against time. The slope of this line divided by the normalizing factor, F, gives the viscosity for purely viscous materials.

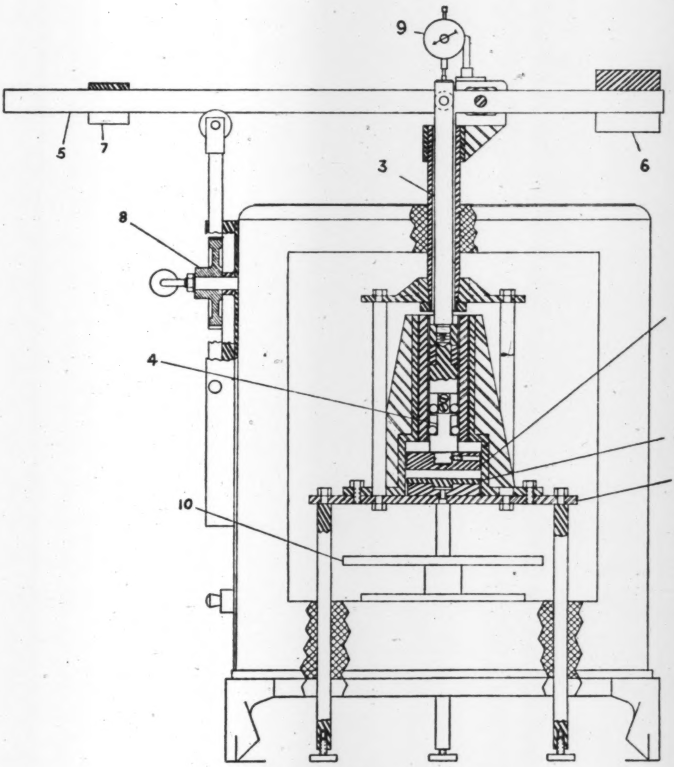

FIG. 23.—Parallel Plate Plastometer.

When the deformation characteristics of the material are functions of elastic, delayed elastic, and viscous properties, a somewhat different situation exists. While, as before, $1/h^4$ is plotted against time, the resulting curve is initially nonlinear but approaches linearity at large values of time (200 to 1000 sec). It has been found that a curve of this type can be conveniently described by a mechanical model consisting of a spring, dashpot, and delayed elastic element (spring and dashpot in parallel) in series (**71**). The deformation-time characteristics for such visco-elastic materials, which are also linear in their response to stress (this includes most thermoplastics in the range of loads experienced in the parallel plate plastometer), are thus described by the equation:

$$F\left(\frac{1}{h^4}\right) = \frac{1}{G_1} + \frac{1}{\eta_3}\,t + \frac{1}{G_2}\,(1 - e^{-t/\gamma_2}) \ldots (46)$$

where:

G_1 = immediate elastic modulus, dynes per sq cm,

η_3 = principal or absolute viscosity, poise,

G_2 = delayed elastic modulus, dynes per sq cm,
γ_2 = retardation time = η_2/G_2, sec, and
η_2 = delayed viscosity, poise.

These constants are readily evaluated from the curve by graphical means (**72**). The absolute viscosity represented by the straight line portion of the $1/h^4$ *versus* time curve, is most frequently employed to define the high temperature characteristics of visco-elastic materials. Absolute viscosities can also be obtained from extrusion-type measurements (**73**) and have been shown to agree well with those from parallel plate measurements (**74**).

References

(1) Poiseuille, *Comptes rendus*, Vol. 11, p. 961 (1840).

(2) Stokes, *Transactions*, Cambridge Philosophical Soc., Vol. 8, p. 287 (1845).

(3) Wiedeman, *Pogg. Ann.* Vol. 99, p. 221 (1856).

(4) Hagenbach, *Pogg. Ann.* Vol. 109, p. 385 (1860).

(5) Barr, "A Monograph of Viscosity," pp. 33, 35 (1931).

(6) Couette, *Annales de Chime et de physique*, Vol. 21, p. 433 (1890); Bousinesq, *Comptes rendus*, Vol. 110, p. 1160 (1890).

(7) Bingham, "Fluidity and Plasticity," p. 67 (1922).

(8) Ubbelohde, *Industrial and Engineering Chemistry*, Analytical Edition, Vol. 9, p. 85 (1937).

(9) A. P. Anderson, K. A. Wright, and R. L. Griffin, "Kinematic Viscometer for Liquid Asphaltic Products," *Industrial and Engineering Chemistry*, Analytical Edition, Vol. 12, pp. 466–468 (1940).

(10) M. R. Cannon and M. R. Fenske, "Viscosity Standardization of Petroleum Lubricating Fractions," *Oil and Gas Journal*, Vol. 33, April 11, 1935, p. 52.

(11) M. R. Cannon and M. R. Fenske, "Simple Viscometer and Auxiliary Equipment for the Accurate Measurement of Viscosity in Fundamental Units," *Oil and Gas Journal*, Vol. 34, April 9, 1936, p. 45.

(12) M. R. Cannon, "Viscosity Measurement," *Industrial and Engineering Chemistry*, Analytical Edition, Vol. 10, p. 297 (1938).

(13) M. R. Cannon and M. R. Fenske, "Viscosity Measurement," *Industrial and Engineering Chemistry*, Analytical Edition, Vol. 13, p. 299 (1941).

(14) Tentative Methods of Test for Kinematic Viscosity (D 445 - 46 T), 1949 Book of ASTM Standards, Part 5, p. 899.

(15) Décharme, *Comptes rendus*, Vol. 74, p. 936 (1872).

(16) E. C. Bingham and H. A. Murray, Jr., "A New Combined Viscometer and Plastomer," *Proceedings*, Am. Soc. Testing Mats., Vol. 23, Part II, p. 655 (1923).

(17) Moness and Giesy, *The Journal of Physical Chemistry*, Vol. 29, p. 1282 (1925).

(18) J. McMillen, *Industrial and Engineering Chemistry*, Vol. 21, p. 1237 (1929).

(19) Pochettino, *Nuevo cimento*, Vol. 8, p. 77 (1914).

(20) Wiggam, *Journal of Rheology*, Vol. 1, p. 48 (1929).

(21) Scott Blair, Industrial Rheology, p. 55 (1938).

(22) Washburn, *The Physical Review*, Vol. 17, p. 277 (1921); Rideal, *The Philosophical Magazine*, Vol. 44, p. 1152 (1922); Herschel, *Industrial and Engineering Chemistry*, Vol. 19, p. 837 (1927).

(23) Bosanquet, *The Philosophical Magazine*, Vol. 45, p. 525 (1923).

(24) A. Pochettino, "Viscosity and Elasticity of Highly Viscous Materials," *Nuevo cimento*, Vol. 8, p. 77 (1914).

(25) P. J. Flory, "Viscosities of Linear Polyesters," *Journal*, Am. Chemical Soc., Vol. 62, p. 1057 (1940).

(26) E. O. Rhodes, E. W. Volkmann, and C. T. Barker, "New Viscometer for Bitumens Has Extended Range," *Engineering News-Record*, November, 21, 1935.

(27) F. S. Stow, Jr., K. H. Horowitz, and J. H. Elliott, "Application of the Rising-Column Capillary Viscometer to the High-Viscosity Range," *Journal of Colloid Science*, Vol. 4, p. 321 (1949).

(28) Bingham, "Fluidity and Plasticity," p. 77 (1922).

(29) Brown and Brouse, Colloid Sym. Monogram, No. V, p. 299 (1927); de Waele, *Journal*, Oil and Color Chem. Assn., Vol. 6, p. 33 (1923); Porter and Gruse, *Industrial and Engineering Chemistry*, Vol. 17, p. 953 (1925); Scott, Blair and Crowther, *The Journal of Physical Chemistry*, Vol. 33, p. 321 (1929).

(30) Arveson, *Industrial and Engineering Chemistry*, Vol. 24, p. 71 (1932).

(31) Merrington, *Nature*, Vol. 152, p. 214 (1943).

(32) Am. Chem. Soc., *Industrial and Engineering Chemistry*, Analytical Edition, Vol. 1, p. 49 (1929).

(33) Stokes, Math. and Phys. Papers, Vol. 2, pp. 10, 3, 56 (1880).

(34) Oseen, Arkiv. f. Mat. Astron. Ock Fysik, Vol. 6, No. 29 (1910); Goldstein, *Proceedings*, Royal Soc., Vol. 123A, p. 225 (1929); Liebster and Schiller, *Physik Zeitschrift*, Vol. 25, p. 670 (1924).

(35) Faxen, *Annalen der Physik*, Vol. 63, p. 581 (1920).

(36) Flowers, *Proceedings*, Am. Soc. Testing Mats., Vol. 14, p. 565 (1914); Hersey, *Journal*, Washington Acad. Sci., Vol. 6, p. 525 (1916); Hersey and Shore, *Mechanical Engineering*, Vol. 50, p. 221 (1928).

(37) Hoeppler, *Proceedings*, World Petroleum Congress, Vol. 2, p. 503 (1933).

(38) Benning and Markwood, *Refrigeration Engineer*, Vol. 37, p. 243 (1939).

(39) Hubbard and Brown, *Industrial Engineering Chemistry*, Vol. 35, p. 1276 (1943).

(40) Markwood, Unpublished.

(41) Hubbard and Brown, *Industrial and Engineering Chemistry*, Analytical Edition, Vol. 15, p. 212 (1943).

(42) Spée, *Transactions*, Chem. Engr. Conf. World Power Conf., Vol. 2, p. 1 (1937); Hoeppler, *Zeitschrift fur technische Physik*, Vol. 14, p. 165 (1933).

(43) M. Segel, "A Method for Determining the Internal Friction of Hard Substances," *Physikalische Zeitschrift*, Vol. 4, p. 493 (1903).

(44) C. J. Mack, "Colloid Chemistry of Asphalts," *Journal of Physical Chemistry*, Vol. 36, p. 2901 (1932).

(45) R. N. Traxler and H. E. Schweyer, "Measurement of High Viscosity—A Rapid Method," *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part II, p. 518 (1936).

(46) M. Couette, "Studies Concerning the Friction of Liquids," *Annales de chimie et de physique*, Vol. 21, No. 6, p. 433 (1890).

(47) E. Humphrey and E. Hatschek, "The Viscosity of Suspensions of Rigid Particles at Different Rates of Shear," *Proceedings*, Physical Soc., London, Vol. 28, p. 274 (1916).

(48) L. E. Gurney, "The Viscosity of Water at Very Low Rates of Shear," *The Physical Review*, Vol. 26, p. 98 (1908).

(49) G. F. C. Searle, *Proceedings*, Cambridge Phil. Soc., Vol. 16, p. 600 (1912).

(50) M. Mooney and R. H. Ewart, "The Conicylindrical Viscometer," *Physics*, Vol. 5, p. 350 (1934).

(51) A. D. Garrison and K. C. Ten Brink, "A Study of Some Phases of Chemical Control in Clay Suspension," *Technical Publication No. 1124*, Am. Inst. Mining and Metallurgical Engrs., p. 20 (1939).

(52) D. V. Lyttleton and R. N. Traxler, "Flow Properties of Asphalt Emulsions," *Industrial and Engineering Chemistry*, Vol. 40, p. 2115 (1948).

(53) E. Stormer, *Transactions*, Am. Ceramic Soc., Vol. 11, p. 597 (1909).

(54) H. Green and R. N. Weltman, "Analysis of the Thixotropy of Pigment-Vehicle Suspensions," *Industrial and Engineering Chemistry*, Analytical Edition, Vol. 15, p. 201 (1943).

(55) H. Green, "High Speed Rotational Viscometer of Wide Range," *Industrial and Engineering Chemistry*, Analytical Edition, Vol. 14, p. 576 (1942).

(56) F. Ross MacMichael, U. S. Patent No. 1,281,042, patented October 8, 1918; and *Journal of Industrial and Engineering Chemistry*, Vol. 7, p. 961 (1915).

(57) T. F. Ford and K. G. Arabian "Measurement of the Deformation Properties of Asphalts," *Proceedings*, Am. Soc. Testing Mats. Vol. 40, p. 1174–1181 (1940).

(58) R. N. Traxler, J. W. Romberg, and H. E. Schweyer, "Rotary Viscometer for Determination of High Consistencies," *Industrial and Engineering Chemistry*, Analytical Edition, Vol. 14, p. 340 (1942).

(59) M. Mooney and R. H. Ewart, "The Conicylindrical Viscometer," *Physics*, Vol. 5, p. 350 (1934).

(60) G. Barr, "A Monograph on Viscometry," Oxford University Press, London (1931).

(61) J. D. Ferry, W. M. Sawyer, and J. N. Ashworth, *The Journal of Polymer Science*, Vol. 2, p. 593 (1947).

(62) T. L. Smith, J. D. Ferry, and F. W. Schremp, *Journal of Applied Physics*, Vol. 20, p. 144 (1949).

(63) W. P. Mason, *Transactions*, Am. Soc. Mechanical Engrs., Vol. 69, p. 359 (1947).

(64) W. P. Mason, *Journal of Colloidal Science*, Vol. 2, p. 147 (1948).

(65) F. R. Innis and E. D. Bailey, Franklin Institute Report No. F-2048, January 3, 1949.

(66) W. O. Baker, J. H. Heiss, and W. P. Mason, "Mechanical Properties of Long Chain Polymers Dissolved in a Solvent," *Transactions*, Am. Physical Soc., January 27, 1949; and more complete paper in process of publication.

(67) W. P. Mason, W. O. Baker, J. H. McSkimin, and J. H. Heiss, "Measurement of the Shear Elasticity and Viscosity of Liquids by Means of Ultrasonic Shear

Waves," *The Physical Review*, Vol. 75, No. 6, March 15, 1949, p. 936.

(68) J. Williams, *Journal of Industrial and Engineering Chemistry*, Vol. 16, p. 362 (1924).

(69) M. J. Stefan, *Akademie der Wissenschiften in Wien*, Abt. 2, Vol. 69, p. 713 (1874).

(70) G. J. Dienes and H. F. Klemm, *Journal of Applied Physics*, Vol. 17, p. 458 (1946).

(71) G. J. Dienes, *Journal of Colloid Science*, Vol. 2, p. 131 (1947).

(72) J. Lipka, "Graphical and Mechanical Computations," J. Wiley and Sons, New York, N. Y. (1918).

(73) R. S. Spencer and R. E. Dillon, *Journal of Colloid Science*, Vol. 3, p. 163 (1948).

(74) G. J. Dienes and F. D. Dexter, *Journal of Colloid Science*, Vol. 3, p. 181 (1948).

APPENDIX

Below is a list of American manufactures of viscometers which has been supplied by the Scientific Apparatus Makers Association.

ENGLER VISCOMETERS:
- American Instrument Co.
- Boekel and Co.

FENSKE VISCOMETERS:
- American Instrument Co.
- Kimble Glass Co.
- Precision Scientific Co.

HOEPPLER VISCOMETERS:
- Fish-Schurman Co.

KINEMATIC VISCOMETERS:
- American Instrument Co.
- Braun Corporation
- Fischer & Porter
- Fisher Scientific Co.
- Koehler Instrument Co.
- Precision Scientific Co.
- Tagliabue Corp.

MACMICHAEL VISCOMETERS:
- Fisher Scientific Co.

RECORDING VISCOMETERS:
- Brabender Corp.
- Brookfield Engineering Labs., Inc.
- Fischer & Porter
- Precision Scientific Co.

REDWOOD VISCOMETERS:
- American Instrument Co.

SAYBOLT VISCOMETERS:
- American Instrument Co.
- Boekel & Co.
- Fisher Scientific Co.
- Precision Scientific Co.

SCOTT VISCOMETERS:
- Boekel & Co.
- Precision Scientific Co.

STORMER VISCOMETERS:
- Arthur H. Thomas Co.

UBBELOHDE VISCOMETERS:
- American Instrument Co.
- Fish-Schurman Co.
- Precision Scientific Co.
- Tagliabue Corp.

ZAHN VISCOMETERS:
- General Electric Co.

REPORT OF COMMITTEE E-2

ON

EMISSION SPECTROSCOPY*

Committee E-2 on Emission Spectroscopy held two meetings during the year: the first at Atlantic City, N. J. on June 28, 1950, and the second in Pittsburgh, Pa., on March 6, 1951. Meetings of the Advisory Committee were held in Atlantic City on June 26, 1950, in Cleveland, Ohio, on October 28, 1950, and in Pittsburgh on March 6, 1951. The membership of the committee is now 124, representing a net gain of 7 members over last year.

The committee organized a Symposium on Chemical Analysis of Inorganic Solids by Means of the Mass Spectrometer in connection with the 1951 Annual Meeting.

The committee recommends no change in the status of its present tentative methods.

Committee E-2 activities were carried out through its subcommittees, all of which showed considerable activity during the year. The committee sponsored the publication of the Report on Standard Samples for Spectrochemical Analysis, 1950, *Technical Publication No. 58B*, which was issued by the Society in October, 1950. A compilation of 48 suggested analytical methods has been prepared in manuscript form, and editing for publication is nearly completed. Publication of the compilation is scheduled in 1952. In addition, in preparation is a manuscript on an extension of the Index to the Literature on Spectrochemical Analysis, covering the years 1946 to 1950 inclusive. This publication is expected to be available in 1952. Progress has been made on the writing of tentative methods by several subcommittees and additional suggested methods are being collected.

The committee organization remains unchanged, except for the assignment of titanium and its alloys to the Subcommittee VII. Spectroscopic methods are being compiled for the analysis of these new materials.

Details of the activities of the subcommittees follow.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Apparatus and Equipment (R. W. Callon, chairman).—The subcommittee, through a group headed by R. G. Russell, has been engaged in the writing of specifications and tolerances for spectrographic equipment. Following a meeting with representatives of equipment manufacturers, a revised report on these subjects was prepared. A study of the quality of photographic materials was continued with the circulation of a report on cooperative testing of an experimental emulsion. Information on the effects of humidity on the speed of photographic emulsions was given to the subcommittee by the Eastman Kodak Co. and was circulated to Committee E-2. A task on surveying the needs of spectrographers for the various types of spectroscopic emulsions was completed by E. K. Jaycox and a report was circulated within Committee E-2.

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*Presented at the Fifty-fourth Annual Meeting of the Society, June 18–22, 1951.

Subcommittee II on Fundamental Methods (R. W. Smith, chairman) is organized into seven groups, all of which have shown progress in their assignments as follows: (*1*) The final report on Specifications for Spark Generators was prepared by R. A. Wolfe, C. H. Corliss, and G. H. Dieke and was distributed to Committee E-2 members; (*2*) a report on Calculations in the Mutual Standard Methods, prepared by J. H. Coulliette, was reviewed and a revised report is being prepared; (*3*) a report by J. Sherman on the Evaluation of Precision and Accuracy in Spectrochemical Analysis was reviewed and a revision is being prepared; (*4*) a report on Photometric Practices prepared by C. Feldman was reviewed in the subcommittee; (*5*) work under D. Fry was continued on Rapid Semi-Quantitative Methods of Analysis and a useful chart prepared comparing the most promising of the methods; (*6*) M. F. Hasler prepared a report on the Internal Standard Method of Spectrochemical Analysis and the report is now being reviewed; (7) H. A. Tuttle is completing a report on Photographic Processing.

Subcommittee III, Editorial (C. L. Guettel, chairman) has been engaged mainly in the final editing of 48 suggested methods for publication by the Society. This task is nearly completed and publication of the compilation is expected late in this calendar year. Revision of some terminology employed in the methods was made and is to be reported to Committee E-2 in the minutes of the June, 1951, Meeting. This committee has carried through a long and arduous job on the compilation of methods and is to be commended for its careful and painstaking work.

Subcommittee IV on Standard Samples and Pure Materials (G. A. Nahstoll, chairman) has been engaged in three main tasks as follows: (*1*) recommendations to the National Bureau of Standards on the compositions and types of desired boron steels and type 347 stainless steels containing tantalum; (*2*) completion of a report on standardization of graphite electrodes by H. M. Bedell; and (*3*) preliminary studies of means of preparing cast iron standard samples.

Subcommittee V on Copper, Nickel, and Their Alloys (E. K. Jaycox, chairman) was engaged in reviewing and revising four suggested methods, which were submitted for publication and bring to a total of eight the methods submitted thus far. One method remains to be revised. The analysis of nickel strip alloys for standardization has progressed but is not complete, since some differences in results remain to be resolved.

Subcommittee VI on Lead, Tin, Antimony, Bismuth and Their Alloys (E. J. Dunn, Jr., chairman) revised two suggested methods which were then distributed to Committee E-2, making a total of eight methods submitted. One method remains to be revised. Tentative methods are in preparation for the analysis of lead, tin, and lead-tin alloys.

Subcommittee VII on Aluminum, Magnesium, and Their Alloys (J. R. Churchill, chairman) completed four suggested methods which were distributed to Committee E-2. A suggested method on the analysis of titanium metal is being reviewed. The first draft of a tentative method for the analysis of aluminum and aluminum alloys was prepared and is being reviewed within the subcommittee.

Subcommittee VIII on Zinc, Cadmium, and Their Alloys (Alan Goldblatt, chairman) has been actively engaged in writing tentative methods of analysis. A revision of Tentative Method of Spectrochemical Analysis of Zinc-Alloy Die Castings for Minor Constituents and Impurities (E 27 – 43 T) on the solution method for the analysis of zinc alloys was prepared. The first draft of a solid pin method for the analysis of zinc alloys was prepared and is now being reviewed.

Subcommittee IX on Ferrous Metals (P. R. Irish, chairman) prepared a tentative method for the analysis of steel which was reviewed within the subcommittee. A revision of the method is being prepared.

Subcommittee X on Non-Metals (R. G. Russell, chairman) distributed ten suggested methods to Committee E-2 and has received an additional method covering the determination of metallic impurities in oils. A group on flame photometry is engaged in collecting methods in this field.

This report has been submitted to letter ballot of the Committee, which consists of 124 members; 94 members returned their ballots, of whom 88 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

B. F. SCRIBNER,
Chairman.

J. H. COULLIETTE,
Secretary.

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This report has been submitted to letter ballot of the Committee, which consists of 124 members; 94 members returned their ballots, of whom 88 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

B. F. SCRIBNER,
Chairman.

J. H. COULLIETTE,
Secretary.

REPORT OF COMMITTEE E-3

ON

CHEMICAL ANALYSIS OF METALS*

The fine work of all the individual members of Committee E-3 on Chemical Analysis of Metals culminated in the issuance of the 1950 Edition of the ASTM Methods of Chemical Analysis of Metals. In brief, there are 476 pages, as compared to 402 pages in the 1946 Edition, and it contains additional features and more adequate coverage of the newer variety of metals and alloys appearing in the metallurgical field.

Committee E-3 held its main meeting at the Annual Meeting of the Society on June 28, 1950, which served not only to coordinate the activities of the divisions and subcommittees but to outline briefly the future plans to improve more fully subsequent editions of the methods book.

It was with regret that the membership was informed of the passing of the first chairman of Committee E-3, Dr. G. E. F. Lundell, chief, Division of Chemistry, National Bureau of Standards. In memoriam, besides the recording in the minutes of the extension of Committee E-3's condolence to his immediate family, it was decided to print a dedication in the 1950 edition of Methods of Chemical Analysis of Metals.

The Advisory Committee, which aids in the establishment of the over-all policies of Committee E-3, held two meetings during the year: on December 1, 1950, at New York, N. Y., and on March 6, 1951, at Pittsburgh, Pa.

The Advisory Committee has been deeply concerned with the proper placement of the work on certain alloys into the several divisions. The newer alloys (for example, nickel-base materials) may sometimes be termed "non-ferrous" alloys, but with slight changes in composition they fall into "ferrous alloys." The Advisory Committee, recognizing the need for a division of Committee E-3 to cover the analysis of metal materials that do not convieniently fall into Division *F* or *N*, created Division *M* on Miscellaneous Metals.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1950 Annual Meeting, Committee E-3 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Method of:

Chemical Analysis of Nickel-Copper Alloys (E 76 - 50 T),

Chemical Analysis for Sulfur in Special Brasses and Bronzes (E 54 - 50 T),

Chemical Analysis of Antimony Metal (E 86 - 50 T),

Chemical Analysis for Aluminum in Zinc-Base Die-Casting Alloys (E 47 - 50 T), and

Sampling Non-Ferrous Metals and Alloys in Cast Form for Determination of Chemical Composition (E 88 - 50 T).

New Tentative Photometric Methods for:

Chemical Analysis of Lead, Tin, Antimony and Their Alloys (E 87 - 50 T).

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* Presented at the Fifty-fourth Annual Meeting of the Society, June 18–22, 1951.

Revision of Tentative Methods for:

Chemical Analysis of Copper-Nickel and Copper-Nickel-Zinc Alloys (E 75 - 49 T).

Revision of Tentative Photometric Methods for:

Chemical Analysis of Aluminum and Aluminum-Base Alloys (E 34 - 49 T),
Chemical Analysis of Magnesium and Magnesium-Base Alloys (E 61 - 46 T) (Redesignated E 35 - 50 T),
Chemical Analysis of Copper and Copper-Base Alloys (E 62 - 46 T), and
Determination of Iron in Slab Zinc (Spelter) (E 64 - 46 T).

Revision of Tentative Recommended Practice for:

Photometric Methods for Chemical Analysis of Metals (E 60 - 46 T).

Tentative Revision of Standard Methods of:

Chemical Analysis of Steel, Cast Iron, Open-Hearth Iron, and Wrought Iron (E 30 - 47), in the form of new tentative methods for beryllium and tin added to Methods E 30 - 48 T.

Revision and Reversion to Tentative of Standard Methods of:

Chemical Analysis of Pig Lead (E 37 - 45),
Chemical Analysis of Lead and Tin-Base Solder Metal (E 46 - 49), and
Chemical Analysis of White Metal Bearing Alloys (E 57 - 49).

Withdrawal of Photometric Method for:

Determination of Bismuth in Pig Lead (E 58 - 45 T),
Determination of Iron in (70-30) Copper-Nickel Alloys (E 63 - 46 T), and
Determination of Iron in Lead- and Tin-Base Alloys (E 67 - 46 T).

These recommendations were accepted by the Standards Committee on September 26, 1950, and the new and revised tentatives, together with the tentative revision of Methods E 30, appear in the 1950 Book of ASTM Methods for Chemical Analysis of Metals.

Activities of Divisions and Subcommittees

Division G on General Analytical Methods (S. E. Q. Ashley, chairman, H. Kirtchik, secretary) held its main meeting, with its subcommittees, at Atlantic City, N. J., in June, 1950. The meeting was concerned primarily with the progress of the subcommittees in preparing material for the 1950 edition of ASTM Book of Methods of Chemical Analysis of Metals. Subcommittee G-1 on Reagents (T. C. Bryson, chairman) prepared the master list of all reagents that appeared in the new edition. G-2 on Apparatus (T. L. King, chairman) reported on the continuance of its work in connection with the standardization of filter papers, and performance standards for balances, photometer filters, and electrolytic apparatus. G-3 on Common Procedures (M. D. Cooper, elected chairman) continued its work on recommendations for common procedures; the work of this subcommittee is to be the subject of discussion at the 1951 Annual Meeting. G-4 on Precision and Accuracy (H. B. Lea, chairman) continues its cooperation with Committee E-11. In addition, a program is being established to evaluate the precision and accuracy that can be established with the methods covered by Division F. Plans made at Atlantic City for a symposium resulted in fruition on September 15, 1950, when a "Symposium on Electro-Analytical Methods" was held at the Mallinckrodt Laboratories of Harvard College, Cambridge, Mass., with J. J. Lingane as host. With S. E. Q Ashley presiding, five main talks were presented.

As a result of the Symposium held at Atlantic City in 1949, a Special Technical Publication No. 98, "Symposium on Rapid Methods for the Identification of Metals," was issued by ASTM in 1950.

Division S on Sampling (J. P. Brull, chairman), as well as its subcommittees S-1 and S-2, held a meeting at the Annual Meeting in Atlantic City in June, 1950. A task force with representatives from Committees B-2, B-5, B-6, and B-7 reviewed the method for sampling of cast non-ferrous metals and alloys and the necessary changes were incorporated under Methods E 88 – 50 T and appear in the 1950 Book. Since the scope of Methods E 88 does not apply to the numerous B-5 specifications for finished castings, it was suggested that any of the present references to Methods E 88 for finished castings, as, for example, in Method E 38, should be deleted and the sampling of the finished castings be left entirely within the casting specifications. Subcommittee S-1, Sampling of Ferrous Alloys (E. J. Dunn, chairman) reviewed and revised methods of sampling ferrotitanium and ferrochromium. Work on screen sizes of samples is progressing. Subcommittee S-2, Sampling of Non-Ferrous Alloys (A. C. Holler, chairman) is in the process of writing a method for sampling of metals for spectrographic analysis in a joint undertaking with E-2. This subcommittee also distributed to the membership a bibliography of sampling of ferrous and non-ferrous alloys. With the resignation of J. J. Aldrich, the new secretary of Division S elected was J. W. Claypool.

Division N on Non-Ferrous Metals (Clarence Zischkau, chairman) met, following the meetings of its subcommittees, on June 26, 1950, at the Annual Meeting of the Society in Atlantic City. The work of testing and editing in connection with the 1950 edition of the methods book was completed by the several subcommittees in time for the publication in November.

Special meetings of local sub-groups have been held as needed, one of which was devoted to the determination of aluminum, a problem common to most of the subcommittees. Experience gained to date in connection with this work suggests the possible desirability of a task group charged with the problem of preliminary development of the method, and operating independent of subcommittee structure.

Subcommittee N-1 on Copper and Its Alloys (R. P. Nevers, chairman) is continuing the work on the development of methods for the analysis of copper-beryllium alloys. Projected work includes the development of methods for the analysis of fire-refined copper and of aluminum as an impurity in copper-base alloys.

Subcommittee N-2 on Lead, Tin, Antimony, Bismuth and Their Alloys (C. L. Luke, chairman) has as proposed work the cooperative testing of a proposed revision of the method for the photometric determination of bismuth in pig lead, and lead- and tin-base alloys, and of a proposed method for the photometric determination of aluminum as an impurity in lead- and tin-base alloys. In addition, the subcommittee has as a project the development of photometric methods for the determination of tin, antimony, and zinc in lead and lead alloys.

Subcommittee N-2 on Aluminum and and Its Alloys (K. C. Braun, chairman) has under consideration the problem of improving the present method for the determination of zinc and the development of photometric methods for the determination of magnesium and silicon in aluminum alloys.

Subcommittee N-4 on Zinc and Its Alloys (R. K. Bell, chairman) has a potential activity program which includes the development of a method for the photometric determination of aluminum in spelter and of improved methods for the determination of aluminum, lead, cadmium, tin, and manganese in zinc-base alloys.

Subcommittee N-6 on Nickel and Its Alloys (C. Sterling, chairman) reports that work is being continued on methods for the analysis of electronic nickel, and has proposed that improvements may be made in the present procedures for the determination of impurities in metallic nickel.

Subcommittee N-7 on Photometric Methods (R. G. Ernst, chairman) has been charged with the project of reviewing existing photometric methods for uniformity, and a consideration of present techniques as to the optimum wavelengths for measurement, etc.

Subcommittee N-9 on Magnesium and Its Alloys (V. A. Stenger, chairman) is planning to test a proposed photometric method for the determination of aluminum in magnesium-base alloys. Consideration is also being given to a revision of the photometric methods for copper and nickel, and for iron. Work is also in progress on the development of methods for the determination of zirconium and of the rare-earth elements.

Division F on Ferrous Metals (Arba Thomas, chairman) held one all-day meeting in June, 1950, during the Annual Meeting of the Society at Atlantic City. The Task Force groups were reorganized, by elements, and sub-chairmen were assigned as follows:

Chairman	*Project*
L. E. Pitzer	Methods for Columbium and Tantalum
E. J. Dunn	Method for Aluminum in Stainless Steels
J. L. Hague	Combustion Sulfur Method for Carbon Steels and Cast Iron
S. Partington	Method for Magnesium in Cast Iron
H. Kirtchik	Method for Zirconium in Steels
M. D. Cooper	Photometric Method for Manganese in Steel
R. M. Fowler	Method for Cobalt in High Alloy Steels
J. J. Furey	Methods for Analysis of Ferrotitanium and Ferroboron

It is anticipated that some of the projects listed will be completed in 1951.

Preliminary work and data on magnesium in cast iron and the photometric method for manganese in steel look promising. More study is required on aluminum in stainless steels.

An effort has been made to invite into the division new members who have had considerable background in the field of the various types of complex high-alloy steels that are being produced in connection with the preparedness program.

The tentative methods for tin and beryllium were approved by letter ballot and are published in the Tentative Methods for Chemical Analysis of Steel (E 30 – 50 T) in the 1950 edition.

The Editorial Subcommittee (S. E. Q. Ashley, chairman) held two meetings during 1950. The first, at the Annual Meeting in June, 1950, was concerned chiefly with final assistance in bringing out the new book. Work continued on a style manual for those engaged in writing new methods, as well as study along the line of revising the general organization of subsequent editions of the book. The second meeting was held at the Chemists' Club in New York City in December, 1950. Among the problems discussed was the lack of uniformity that exists among various national organizations on the subject of analytical nomenclature. Committee E-3 is to work closely with Committee E-8 and possibly other groups to hasten the progress towards unification. A subcommittee was formed to work with Committee E-8 on the proposed "Recommended Practices Manual." Another topic that the Editorial Subcommittee is to consider is the method of expressing sample weights.

This report has been submitted to letter ballot of the committee, which consists of 123 members; 76 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

J. W. STILLMAN,
Chairman.

H. KIRTCHIK,
Secretary.

REPORT OF COMMITTEE E-4

ON

METALLOGRAPHY*

Committee E-4 on Metallography and most of its subcommittees held meetings on March 5 and 6, 1951, at The Committee Week gathering of the Society in Cincinnati, Ohio.

Membership of the committee continues to grow, and Committee E-4 now has a roster of 96 members.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the Annual Meeting, Committee E-4 presented to the Society through the Administrative Committee on Standards the recommendation that the Methods of Classification of Ferrite Grain Size in Steels be published as tentative. This recommendation was accepted by the Standards Committee on September 26, 1950, and the new tentative appears in the 1950 Supplement to Book of ASTM Standards, Part 1, bearing the designation E 89 – 50 T.

NEW TENTATIVES

A Proposed Method for Estimating the Average Grain Size of Non-Ferrous Metals and Their Alloys Other Than Copper and Copper-Base Alloys and a Proposed Recommended Practice for Thermal Analysis of Metals and Alloys intended to replace the Standard Recommended Practice for Thermal Analysis of Steel (E 14 – 33) have been submitted to the Administrative Committee on Standards for publication as tentative.[1]

ADOPTION OF TENTATIVE AS STANDARD

The committee recommends that the Recommended Practice for Determining the Inclusion Content of Steel (E 45 – 46 T)[2] be approved for reference to letter ballot of the Society for adoption as standard.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends that the following six tentatives be continued without revision:

Tentative Methods of:

Preparation of Micrographs of Metals and Alloys (E 2 – 49 T),
Preparation of Metallographic Specimens (E 3 – 46 T),
Preparing Quantitative Pole Figures of Metals (E 81 – 49 T), and
Determining the Orientation of a Metal Crystal (E 82 – 49 T).

Recommended Practices for:

Identification of Crystalline Materials by the Hanawalt X-Ray Diffraction Method (E 43 – 49 T), and
Dilatometric Analysis of Metallic Materials (E 80 – 49 T).

The recommendation appearing in this report has been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Selection and Preparation of Samples (F. C. Hull, chairman) is recommending that E 3 –

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* Presented at the Fifty-fourth Annual Meeting of the Society, June 18–22, 1951.

[1] See Editorial Note, p. 485.

[2] 1949 Book of ASTM Standards, Part 1.

[3] The letter ballot vote on this recommendation was favorable; the results of the vote are on record at ASTM Headquarters.

46 T on Preparation of Specimens be retained in their present form for the time being. Recognizing the many advances in technique, as well as the new metals and alloys now in use, the subcommittee is engaging in a thorough revision and elaboration of E 3 to bring the recommended methods completely up to date.

Subcommittee II on Definitions (R. S. Busk, chairman) regrets the pressure of work which made it necessary for P. A. Beck to relinquish his chairmanship after giving so much impetus to the definition work.

R. S. Busk has taken over as chairman, and the group is currently holding its second meeting of the year, working diligently on compiling and editing the many hundreds of approved metallographic terms.

Subcommittee III on Nomenclature (P. A. Beck, chairman) is currently assembling the many suggestions concerning phase nomenclature into one report for the purpose of detailed study and future planning of program.

Subcommittee IV on Preparation of Micrographs has a new chairman, Sidney Poole, who replaces its capable and long-time leader L. V. Foster, whose new assignments prevent his continuing with this group. The subcommittee is recommending the retention of Methods E 2 in their present form.

Due to the outstanding success of the photomicrographic exhibits and the high value of achievement which they represent, the subcommittee is already making plans for the 1952 display and will make special efforts to encourage participation by students.

Subcommittee V on Microhardness (Alexander Gobus, chairman) has had a busy year working in collaboration with Committee E 1 in preparing a Proposed Method of Test for Diamond Pyramid Hardness of Metallic Materials, which is currently being submitted for letter ballot.

Subcommittee projects on tests of various types of machines and round-robin tests by a number of laboratories will be completed and form the basis of discussion for the group's second meeting this year. Also, the terms and definitions are scheduled for completion at this meeting.

Subcommittee VI on X-Ray Methods (W. L. Fink, chairman) has been engaged in a review of Tentative Methods E 43, E 81, and E 82. It was found that E 43 - 49 T will require major changes which cannot be completed and approved prior to the June Meeting. It is therefore planned to continue this tentative in effect without change for one more year. The other two tentative methods require very minor changes which are being submitted to letter ballot.

Work is progressing in two different laboratories on the appraisal and the improvement of the Schultz method for determining the central part of pole figures. The work has not yet reached a stage where this method can be included in the tentative Method E 81 - 49 T.

Subcommittee VII on Methods of Thermal Analysis, under its new chairman, D. I. Finch, has been engaged for the last two years in writing a proposed tentative Recommended Practice for Thermal Analysis of Metals and Alloys to replace Standard E 14 - 33. The new tentative was completed and approved by both the subcommittee and Committee E-4 prior to the Spring Meeting in Cincinnati. At that meeting a few editorial changes were made.

Subcommittee VIII-A on Austenitic Grain Size (M. A. Grossman, chairman) is recommending that the Austenitic Grain Size Standard E 19 - 46 be submitted to the American Standards Association for approval as American Standard.

Subcommittee VIII-C on Non-ferrous Grain Size (C. A. Samans, chairman) has occupied itself with two major activities: (*1*) revision of E 79 - 49 T, Tentative Methods for Estimating the Average Grain Size of Wrought Copper and the Copper-Base Alloys, and (*2*) completion of the proposed Tentative Methods for Estimating the Average Grain Size of Non-ferrous Metals and their Alloys, other than Copper and Copper-Base Alloys. After due consideration, it was voted to continue E 79 - 49 T as tentative without change.

The new "Non-ferrous Grain Size Standard" has been completed. Differences found in the individual sets of grain size photographs used by various companies have been corrected so that the new tentative includes standard grain size photographs for both twinned and untwinned materials ranging from ASTM No. 2 to ASTM No. 10 in increments $\frac{1}{2}$ ASTM grain size number. To facilitate use, the average grain "diameter" is given in both inch and millimeter units. This new tentative also includes procedures for using the standard set of grain-size micrographs for estimating macro grain sizes up to the order of 1 in. (25 mm) average grain "diameter." The macro grain size system and the micro grain size system overlap in the range 0.020 in. (0.050 mm) to 0.007 in. (0.180 mm).

Despite the pressure of new assignments, Mr. Samans has remained as chairman of the group throughout the trying circumstances incident to these outstanding achievements in establishing uniform methods of grain-size determinations in the non-ferrous field. Completing a job "well done," he is turning the chairmanship over to H. P. George.

Subcommittee IX on Inclusions (Samuel Epstein, chairman) is recommending that E 45 - 46 T be adopted as standard.

Subcommittee X on Decarburization (J. J. B. Rutherford, chairman) is continuing its participation in the Joint SAE-AISI-ASTM Division XXX group, working in the direction of suitable definitions and methods. There are no recommendations as yet.

Subcommittee XI on Electron Microstructure of Steel (D. M. Teague, chairman) is currently studying the bainite structures in detail. The first progress report on the study of steel microstructure with the electron microscope,[4] brought out the fact that two distinct structures may be observed. Information is sought regarding the transformation range over which the lower temperature, striated-needle type of bainite is formed, as well as the range and appearance of the upper bainite structures. Results of the investigation of these hitherto incompletely resolved structures will be submitted for publication under the auspices of the ASTM.

Two additional laboratories are now participating in the electron microscope research program. Each member is preparing electron micrographs of a series of bainite specimens transformed at 50 deg intervals from 500 to 850 F. In addition to studying the microstructures with specified etchants and replica techniques, other new procedures will be examined by individual members.

Representatives of this committee have been appointed to cooperate with Subcommittee II in the preparation of a list of terms and definitions in the field of metallurgical electron microscopy.

This report has been submitted to letter ballot of the committee, which consists of 96 members; 66 members returned their ballots, of whom 66 voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

L. L. WYMAN,
Chairman.

MARY R. NORTON,
Secretary.

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[4] *Proceedings*, Am. Soc. Testing Mats., Vol. 50, p. 444 (1950).

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee E-4 presented to the Society through the Administrative Committee on Standards the recommendation that the Recommended Practice for Thermal Analysis of Steel (E 14 – 33) be revised and reverted to tentative. This recommendation was accepted by the Standards Committee on July 10, 1951, and the revised recommended practice appears in the 1951 Supplement to Book of ASTM Standards, Parts 1 and 2, under the new title "Tentative Recommended Practice for Thermal Analysis of Metals and Alloys."

On August 21, 1951, the Tentative Method for Estimating the Average Grain Size of Non-Ferrous Metals and Their Alloys Other Than Copper and Copper Base Alloys (E 91 – 51 T) was accepted by the Administrative Committee on Stnadards. The tentative method appears in the 1951 Supplement to Book of ASTM Standards, Part 2.

REPORT OF COMMITTEE E-7

ON

NON-DESTRUCTIVE TESTING*

Committee E-7 on Non-Destructive Testing met at Atlantic City, N. J., on June 28, 1950, in conjunction with the Annual Meeting of the Society. Numerous Executive Council and subcommittee meetings have been held since.

The membership of the committee is is now 98, including 3 consulting members.

TENTATIVES CONTINUED WITHOUT REVISION

In accordance with recommendations of special subcommittees, it is recommended that the Tentative Industrial Radiographic Terminology for Use in Radiographic Inspection of Castings and Weldments (E 52 – 49 T) and Tentative Industrial Radiographic Standards for Steel Castings (E 71 – 47 T) be continued for one more year without revision.

GENERAL ACTIVITIES OF THE COMMITTEE

Publication of the two collections of papers, on Radiography and Ultrasonic Testing respectively, referred to in the 1950 Report of the committee, was completed during the year. In addition, the papers presented at the symposium on the Rôle of Non-Destructive Testing in the Economics of Production, sponsored by the committee during the 1950 Annual Meeting, have been collected into a separate volume and published. These publications are now available as ASTM Special Technical Publications Nos. 96, 101, and 112 respectively.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Recommended Radiographic Practice (H. E. Seemann, chairman) submitted the initial draft of its Proposed Tentative Recommended Practice for Radiographic Testing to the full committee membership for comment and trial. Revision of the material, on the basis of comments received, is in progress. It is planned to have the final draft available to submit to the committee for action at the Annual Meeting.

Subcommittee II on Radiographic Standards for Steel Welds (Alexander Gobus, chairman) has received radiographs of samples prepared by three different members and anticipates further contributions before the Annual Meeting, at which time it is hoped that choice of a final set of samples covering manual electric arc welding, at least, can be made.

Subcommittee III on Magnetic Particle and Penetrant Testing (H. Migel, chairman) has prepared a preliminary procedure for magnetic particle testing by dry powder method which may be ready for final action by the time of the Annual Meeting. Further plans also include developing a set of standard indications which may be revealed by the procedure finally adopted.

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* Presented at the Fifty-fourth Annual Meeting of the Society, June 18–22, 1951.

Subcommittee IV on Technical, Economic and Application Data (R. C. McMaster, chairman), which was responsible, with H. H. Lester, for the Symposium at the 1950 Annual Meeting, publication of which is referred to above, has as its 1951 project a technical meeting or papers session to follow the annual meeting of Committee E-7. Several papers on technical developments in non-destructive testing are planned.

Subcommittee V on Radiographic Procedure Specifications (C. H. Hastings, chairman) held its organization meeting in March and defined its objective as the preparation of "a definite and workable specification by means of which significant radiographic inspection data can be assured." Preliminary approach is to be *via* the study of radiographic procedure specifications now in existence and the correlation of these, with comments by the subcommittee, as a means of developing the outlines of a suitable preliminary document.

Subcommittee VI on Ultrasonic Testing (J. C. Smack, chairman) has started work on five separate projects, including procedures for reflection and resonance methods, development of calibration blocks for the methods, and a glossary of terms. It is hoped that some initial work on all five can be accomplished by the time of the Annual Meeting.

PLANS FOR 1952

It has been proposed that the committee sponsor an International Symposium on Non-Destructive Testing in conjunction with the Fiftieth Anniversary Program in 1952. This possibility is being explored.

This report has been submitted to letter ballot of the committee, which consists of 95 voting members; 67 members returned their ballots, of whom 65 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

J. H. Bly,
Chairman.

D. T. O'Connor,
Secretary.

REPORT OF COMMITTEE E-9

ON

FATIGUE*

Organizational changes have been made during the past year to improve the effectiveness of the operation of Committee E-9. The Advisory Committee has been enlarged to include the subcommittee chairmen, resulting in two new members, T. J. Dolan and W. T. Lankford. Subcommittee I on Research (H. F. Moore, chairman) and Subcommittee III, Survey (T. J. Dolan, chairman) remain unchanged. Subcommittee II on Papers (W. T. Lankford, chairman) has been reconstituted. A Subcommittee IV on Large Machines and Test Correlation (J. M. Lessells, chairman) has been formed.

A considerable number of papers on fatigue have been screened during the year; of these the following 13 papers have been accepted:

"The Fatigue Test as Applied to Lead Cable Sheath," G. R. Gohn and W. C. Ellis, Bell Telephone Laboratories, Inc.[1]

"The Influence of Frequency on the Repeated Bending Life of Acid Lead," J. F. Eckel, General Electric Co.[2]

"Fatigue Tests in Axial Compression," N. M. Newmark, R. J. Mosborg, W. H. Munse, and R. E. Elling, University of Illinois.[3]

"Planning and Interpretation of Fatigue Tests," A. M. Freudenthal, Columbia University.[4]

"On the Statistical Nature of Fatigue," F. B. Stulen, Curtiss Wright Corp.[4]

"Statistical Analysis of Fatigue Data," Robert Plunkett, Hughes Tool Co.[4]

"Fatigue Strength of Ball Bearing Races and Heat Treated 52100 Steel Specimens," Haakon Styri, SKF Industries Inc.[5]

"The Influence of Surface Roughness on the Fatigue Life and Scatter of Test Results of Two Steels," P. G. Fluck, University of Wisconsin.[6]

"Effect of Residual Stress on the Fatigue Strength of Notched Specimens," D. Rosenthal and G. Sines, University of California.[7]

"Damping, Elasticity, and Fatigue Properties of Temperature Resistant Materials," B. J. Lazan and L. J. Demer, University of Minnesota.[8]

"Damping, Fatigue and Dynamic Stress-Strain Properties of Mild Steel," B. J. Lazan and T. Wu, University of Minnesota.[9]

"Laboratory Evaluation of Materials for Marine Propulsion Gears," M. R. Gross, U. S. Naval Engineering Experiment Station.[10]

"Electronically Excited Resonance-Type Fatigue Testing Equipment," T. J. Dolan, University of Illinois.[11]

Three Fatigue Sessions are being held at the Annual Meeting, one of the sessions dealing with the Statistical Aspects of Fatigue.

The Manual on Fatigue Testing (*STP No. 91*) is being well received in the professionals areas which it was intended to serve. Over 1000 copies were sold during the first year.

Committee E-9 has participated in the Project Squid Conference at Washington, D. C., May 24, 1950 (proceedings unclassified) and in the Fatigue and Fracture of Metals Conference at Massachusetts Institute of Technology, June 19-22, 1950.

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* Presented at the Fifty-fourth Annual Meeting of the Society, June 18–22, 1951.

[1] See p. 721.

[2] See p. 745.

[3] See p. 792.

[4] Published in separate publication, *STP No. 121*, see p. 583.

[5] See p. 682.

[6] See p. 584.

[7] See p. 593.

[8] See p. 611.

[9] See p. 649.

[10] See p. 701.

[11] Published in ASTM BULLETIN, No. 175, July, p. 60 (TP 150).

Committee E-9 has supplied information on fatigue testing machines to M. Hempel of the Institut für Eisenforschung (Düsseldorf) to be used in the new edition of the Handbook on Material Testing (in German).

Nomenclature and symbols are under discussion between Committee E-9 and other committees having areas of common interest.

Exchange of information on fatigue problems with the British MERB Committee is active through H. J. Gough, corresponding member.

The Survey Subcommittee (T. J. Dolan, chairman) has collected from the membership of Committee E-9 eleven problem statements for the Administrative Committee on Research. These statements average approximately one typed page each and include: (*a*) Problem; (*b*) Present state of knowledge; (*c*) Questions to be answered; and (*d*) Introductory references. The subjects are:

(*1*) Fatigue properties of fully hardened steels above Rockwell C-45. (fatigue strength, notch sensitivity, effect of residual stresses).
(*2*) Relation between stress history and fatigue damage (overstressing, understressing, rest periods).
(*3*) Effect of local plastic flow on notch sensitivity.
(*4*) Character and statistical nature of fatigue damage.
(*5*) Fatigue properties of timber fasteners under repeated loads (joints made with nails, bolts, screws, connectors).
(*6*) Evaluation of fatigue notch-sensitivity index.
(*7*) Effect of size and shape of a member upon fatigue behavior.
(*8*) Effect of shape of notch.
(*9*) Fatigue strengths of metals subjected to combined stresses (various bi-axial combinations).
(*10*) Study of crack genesis and growth.
(*11*) Effect of various factors on fatigue life (surface finish, surface treatment, frequency, overstressing, understressing,—note scatter in each case).

It is hoped that the statements will be especially useful to universities and government laboratories in planning research in the fatigue field.[12]

The Survey Subcommittee has taken initial steps to formulate a plan for providing a current fatigue literature reference service.

Subcommittee IV has been formed as a result of extensive discussion at the E-9 meeting held in Cincinnati, March 5, 1951. Fatigue failures of tailshafts of ships and fatigue problems in aircraft design have increased our interest in the fatigue of large members. It is hoped that the Navy and Timken fatigue machines can be used at least to a limited extent on E-9 sponsored tests.

Allied to the problem of testing large members is the question of appropriate statistical analysis. This has been discussed at considerable length during the past year without arriving at a stage where definite recommendations can be made; further information will be forthcoming at the Session on Statistical Aspects of Fatigue.

This report has been submitted to letter ballot of the committee, which consists of 41 members; 32 members returned their ballots, all of whom have voted affirmatively.

Respectively submitted on behalf of the committee,

R. E. PETERSON,
Chairman.

O. J. HORGER,
Secretary.

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[12] Until available in published form, a copy of any particular statement (or statements) can be obtained by writing to T. J. Dolan, Talbot Laboratory, University of Illinois, Urbana, Ill.

REPORT OF COMMITTEE E-11

ON

QUALITY CONTROL OF MATERIALS*

The Society has announced the publication of the ASTM Manual on Quality Control of Materials which has been prepared by Committee E-11. This is a revision and replacement of the Society's Manual on Presentation of Data whose main section and two supplements were first published respectively in 1933 and 1935. The manual contains three parts: Section 1 which discusses the application of statistical methods in the presentation of data, Section 2 which discusses the problem of presenting limits to indicate the uncertainty of the average X, of a unique sample of n observations and Section 3 which describes the "Control Chart" method of analysis and presentation of data.

The Manual was prepared by two task groups of Committee E-11, No. 1, which prepared Sections 1 and 2 and No. 2 which prepared Section 3. Task Group No. 1 was headed by Mr. R. F. Passano with Messrs. A. C. Holman, J. T. MacKenzie and H. F. Dodge as members. Task Group No. 2 was headed by Dr. A. E. R. Westman with Messrs. A. I. Peterson, H. G. Romig, L. E. Simon and H. F. Dodge as members. The task groups were greatly assisted in their work by Miss M. N. Torrey of the Bell Telephone Laboratories. A cooperate relationship was set up between the ASTM and the American Society for Quality Control with respect to this work (Committee E-11 and ASQC Standards Committee) whereby the latter may, on behalf of ASQC, consider endorsing individual sections of the new ASTM Manual on Quality Control of Materials. The new manual has had the advantage of this joint consideration and its three sections carry the endorsement of the ASQC.

Task groups of Committee E-11 have been working on specific projects aimed to promote the knowledge of statistical quality control methods and their application to specifications and methods of test. Task Group No. 7 on Sampling of Bulk Materials is holding a symposium on the sampling of bulk materials at the Annual Meeting of the Society in Atlantic City. The purpose of this group is to study and report on the problems of sampling materials that occur in bulk form or in packages with the aim of estimating, at minimum cost, measurable characteristics of a quantity of material in order to get some prescribed limit of error with an assigned probability.

Task Groups on surveying ASTM sampling plans, recommended practices for planning of interlaboratory tests, procedures for determining the number of tests for a desired precision of an average, the fitting of curves for a linear relationship, and the study of problems concerned with the use of the terms "precision" and "accuracy" are in various stages of completion of their work.

Committee E-11 has given assistance to other ASTM committees and members on problems covering special phases of application of statistical methods to ASTM problems.

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* Presented at the Fifty-fourth Annual Meeting of the Society, June 18–22, 1951.

This report has been submitted to letter ballot of the committee, which consists of 23 members; 20 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

H. F. DODGE,
Chairman.

O. P. BECKWITH,
Secretary.

REPORT OF COMMITTEE E-12

ON

APPEARANCE*

Committee E-12 on Appearance has held two meetings during the past year; in Atlantic City and Washington, D. C.

The membership of the committee has grown to a total of 61, with official representatives designated by 16 ASTM technical committees.

This committee represents ASTM in the Inter-Society Color Council and American Standards Association Committees A13 (on Scheme for Identification of Piping Systems) and Z58 (on Standardization of Optics).

The expanding interest in this committee's scope of activity was clearly indicated by a registered attendance of 83 at the full day two-session program sponsored by Committee E-12 in Washington, D. C., on February 27, 1951, with an estimated total attendance of 150. The formal papers presented at this meeting concerned the theme: "Appearance, Its Description, Measurement and Specification,—A Study of What is Being Done by Others and What Needs to be Done." Reports were presented by representatives of Inter-Society Color Council, American Standards Association, Optical Society of America, Illuminating Engineering Society, International Commission on Illumination, and American Association of Textile Chemists and Colorists.

Reports on the past experiences and present needs of the following ASTM technical committees as they relate to the appearance properties of materials were also presented: Committees C-21 on Ceramic Whitewares, C-22 on Porcelain Enamels, D-1 on Paints and Protective Coatings, D-4 on Road Materials, D-6 on Paper, D-12 on Soaps and Other Detergents, D-20 on Plastics, D-2 on Petroleum Products and Lubricants, D-13 on Textiles, D-17 on Naval Stores, and D-19 on Industrial Water.

Records were made of the presentations and it is planned that the Advisory Committee will carefully study these with the view to classifying the problems and take immediate steps in organizing work groups for their study.

A procedure for efficient consideration of inquiries was formulated whereby Committee E-12 will cooperate with the technical committee having most direct interest in a problem. The cooperation of this committee has been specifically offered to several committees whose scope of work includes appearance problems which have been brought to the attention of this committee.

This report has been submitted to letter ballot of the committee, which consists of 62 voting members; 36 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

M. Rea Paul,
Chairman.

Daniel Smith,
Secretary.

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* **Presented at the Fifty-fourth Annual Meeting of the Society, June 18–22, 1951.**

TECHNICAL PAPERS

With Discussions

EDGAR MARBURG LECTURE

THE PURPOSE of the Edgar Marburg Lecture is to have described at the Annual Meetings of the American Society for Testing Materials, by leaders in their respective fields, outstanding developments in the promotion of knowledge of engineering materials. Established as a means of emphasizing the importance of promoting knowledge of materials, the Lecture honors and perpetuates the memory of Edgar Marburg, first Secretary of the Society.

CORROSION TESTING[1]

TWENTY-FIFTH EDGAR MARBURG LECTURE

BY F. L. LAQUE[2]

The American Society for Testing Materials started to devote a good deal of attention to corrosion, and particularly corrosion testing, at the time Edgar Marburg was contributing so much to the ultimate success of this organization. The fields of engineering and the applications of materials represented by the members of the Advisory Committee on Corrosion will support the statement that at this time corrosion is a matter of some concern in a larger percentage of the Society's activities than any other subject that engages the attention of its members. Twelve committees of the Society are now represented on its Advisory Committee on Corrosion. It is, therefore, fitting that, in this addition to the record of the accomplishments which have been fruits of the labor of the man whose memory we are honoring on this occasion, we should provide a review of the Society's activities in corrosion testing and a picture of where we stand in this important field. This will supplement the more extensive treatment of the subject in the Symposium on Corrosion Testing Procedures held by the Society in 1937.[3]

This Society has concerned itself primarily with the performance of materials when subjected to corrosive action rather than with the theoretical aspects of corrosion processes. The substantial contributions to corrosion theory that may be found in the papers and reports presented at Society meetings have, in general, been incidental to the development and appraisal of testing methods, to the interpretation of results of the Society's test programs, and to the establishment of proper limits for constituents that may either improve or impair the corrosion resistance of materials covered by the Society's specifications.

Corrosion problems can be divided roughly into two classes—those of general interest and those of particular interest. In making this distinction, the author is fully aware that every practical corrosion problem is a specific one and requires careful consideration in the application of available data to its solution. A problem of general interest may be defined as one that is related to a corrosive environment that may be encountered by a great many people, such as the atmosphere, or to a material that is in common use in many environments. A problem of particular interest is one that may be encountered in the extreme case in only a single instance, such as in the concentration of some rare chemical in a certain type of evaporator or the use of some rare material in some limited way.

Quite properly, the Society's own investigations of corrosion have been confined to environments of general interest, while its contributions to the solution of problems of particular interest have stemmed from its recommended practices for carrying out corrosion tests that may be used in finding a solution to any corrosion problem.

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[1] Presented on June 29, 1951, before the Fifty-fourth Annual Meeting of the Society, Atlantic City, N. J.

[2] In Charge of Corrosion Engineering Section, Development and Research Division, The International Nickel Co. Inc., New York, N. Y.

[3] Symposium on Corrosion Testing Procedures, Am. Soc. Testing Mats. (1937). (Symposium issued as separate publication, *STP No. 32.*)

The Society has also devoted considerable attention to tests that may be used as performance tests for specification purposes or to measure some peculiar quality of a material that may be of special interest. Of necessity, the conditions under which such tests are carried out have had to be standardized and prescribed rather rigidly. As a consequence of this, it follows that the results of such tests will apply directly only to those very limited and rarely encountered conditions of use that match those of the standardized tests. Failure to recognize this fact and the understandable desire to get information quickly and easily have resulted in occasional misapplications of the tests. Resulting disappointments have been due not so much to deficiencies in the tests themselves as to abuses in the use of them.

Similarly, in the case of the tests in environments of general interest, such as in different atmospheres, it has not been practical in the placement of test specimens to arrange for them to encounter the natural corrosive environments under all the conditions that may be met by a particular structure or component at a particular location. Performance is affected by the degree of shelter from the sun and the rain, the direction of the wind with respect to peculiar sources of atmospheric "pollution," the nature and extent of such pollution, the amount of rainfall and dew, the orientation of the exposed surfaces, and so on, all of which cannot be duplicated in a practical manner by the exposure of a group of specimens in a prescribed position at a limited number of test sites.

Recognition of these limitations and the basis for them in the direct application of test results does not destroy the value of the results. Instead, it points the way to their profitable use not only in selecting a suitable material or protecting one in a proper way, but also in designing or arranging the materials in use so that they will have the best possible chance of resisting the corrosive action they may encounter.

The natural human tendency to wish to get information quickly extends to information on corrosion and is the basis for the numerous attempts that have been made, and are still being made, to find some methods of "accelerated" testing that will give the same results in a few hours or a few days as would be secured by exposure for months or years under the natural conditions of use to which the test results are to be applied.

The desired "acceleration" may be obtained by modifying one or more of the environmental factors so as to speed up the rate of attack. This may take the form of increasing the temperature, increasing the concentration of some constituent of the environment or corrosive medium, adding some corrosive constituent not normally encountered, substituting a corrosive medium believed to be much more corrosive than the one of interest, applying electrical current from an external source so as to speed up the normal electrochemical processes of corrosion (**1**),[4] or inducing greater electrochemical corrosion by making the test piece the anode of a galvanic couple (**2**).

Along the same line, is the use of some quickly made observation, such as the potential of a metal with respect to some reference electrode, as a criterion of corrosion resistance. This includes the use of potential measurements of specimens under the influence of currents applied at different voltages to indicate what has been called the "breakdown potential" on passive metals, such as stainless steels and tin (**3**). This technique is useful in comparing different alloys with respect to the stability of their passive films and in studying the influence of environmental factors such as temperature on the breakdown of passivity (4).

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[4] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 579.

Similarly, the rate and extent of recovery of passivity after deliberate reduction or destruction of passive or other corrosion product films may be indicated by potential measurements (5, 6) which will permit at least a qualitative comparison of different alloys as to the facility with which they can be made passive as a result of different treatments and in media of particular interest. This method can be used also to study effects of changes in the nature of the corrosive medium on the incidence and extent of passivity.

Not quite the same as an accelerated test is the securing of data on rates of corrosion under natural or simulated exposure conditions by means of very short tests and then extrapolating these very short-time tests results to predict behavior over a very long period of exposure.

During the course of its existence, the Society has concerned itself with many of these "accelerated" tests and has done a great service in demonstrating their serious limitations. It is true that some of the test methods that have been developed by the Society can be misapplied in carrying out "accelerated" tests, but this is not so much the fault of the Society as it is of those who use the tests improperly in their preference for a quick result rather than the correct one.

Another substitute for long-time tests such as the Society has undertaken in natural environments is the use of data from service records or experience. This information is undoubtedly of great value when interpreted correctly and applied properly, and especially when it can be tied in with results of controlled test programs. But such data frequently suffer from the disadvantage in interpretation and application that not enough is known or can be discovered about the exact circumstances of the use that provides the service record with respect to several of the factors that may affect corrosion or, if this information is available, the influence of a predominant factor may not be appreciated or given proper recognition.

In the course of this lecture, the author will draw upon data from the Society's investigations and from his own experience, particularly in the study of corrosion in marine environments, to illustrate some of the facts and principles that have been touched upon in these introductory remarks.

Atmospheric Corrosion Studies

At the time the Society began to devote attention to corrosion, there was a great deal of controversy over the relative merits of the different kinds of iron and steel in resisting corrosion—particularly atmospheric corrosion. Advocates of the different products were accustomed to cite case histories to support claims of superiority of one kind of iron and steel over another. When data from tests were presented either to bolster or undermine the conclusion being drawn from service records, such results as were unfavorable to a particular class of material were challenged by the advocates of such materials either by citing apparently contrary data or experiences or by the introduction of polemic arguments based largely on assertions as to the inherent corrosion-resisting qualities of the materials under discussion.

When different kinds of iron or steel were joined in the same structure and were corroded to different extents, the proponents of the less attacked material would use the case as evidence of its superiority while the defenders of the more attacked might attribute its failure to some peculiar action such as galvanic corrosion resulting from the combination of the dissimilar materials in the same structure. (In view of present knowledge *re* the limited distance through which galvanic action can extend in atmospheric exposures, it seems unlikely that galvanic effects were responsible in these cases. It is more likely that differences in exposure conditions with respect to the entrapment of moisture and rates of drying, as well as the presence or absence of residual elements such as copper, phosphorus, and sulfur were more important.)

And, then, there were always questions as to the extent to which any particular specimen of iron or steel in service should be considered as having been truly representative of its kind and, therefore, satisfactory for measuring over-all ability to resist corrosion.

The several discussions of this sort never led to any clean-cut conclusions. While the test programs that have been undertaken by the ASTM have not put an end to this sort of controversy, they have at least provided a body of data based on specimens of known composition and history, selected to be representative of their kind and exposed under the same conditions at the same time and place and thus have eliminated many of the bases of the arguments that have obscured the interpretation of case histories.

THE ACID TEST

During the course of these early arguments, some attention was given to the development of an acid test which some hoped might serve as an accelerated test to predict the durability of the different kinds of iron and steel under conditions of atmospheric exposure (7). This test was simple enough. It involved only measuring the loss in weight of a specimen prepared in a certain manner and exposed to corrosion by a 20 per cent solution of sulfuric acid for 1 hr at 15 C (59 F).

Results of acid tests were reported by Committee U (now Committee A-5) in the *Proceedings* for 1908. The data showed great variations amongst the different kinds of iron and steel, with the Bessemer steels, as a class, being attacked to the greatest extent and the open-hearth steels and irons least. The wrought irons and charcoal irons, with a few exceptions, were intermediate. It seems likely that the major difference observed was due primarily to the fact that the high phosphorus content of the Bessemer steels increased their attack by sulfuric acid (even though phosphorus adds considerably to resistance to atmospheric corrosion).

At the very time that this acid test was first considered, some members of Committee U had misgivings as to its value. The following is quoted from the 1908 report:

> "Since the life of some materials is limited by the localized action known as pitting, it is the opinion of the committee that too much dependence should not be placed on the acid test as indicating the relative life of the metal in service."

Of greater importance than the reason cited by the committee is the fact that the acid test places greatest weight on the corrodibility of the metal while we know, now, that the principal determinant of the atmospheric durability of steels is the protective value of the rust films that they form. The mechanisms by which the constituents of steels and irons influence the protective qualities of their rusts were discussed in detail by H. R. Copson (8) before this Society in 1945. Copson's brilliant elucidation of the ways in which certain constituents of steel may influence their durability in atmospheric exposure by changing the protective qualities of their rusts rather than by any more direct effects on the corrodibility of the steels themselves might have been anticipated many years earlier if the suggestion made by one of the early investigators had been followed more diligently. In discussing a paper by H. M. Howe and Bradley Stoughton (9) in 1908, G. W. Thompson said, "I would suggest . . . that this line of study, the composition of rust would be exceedingly profitable in connection with the corrosion of iron."

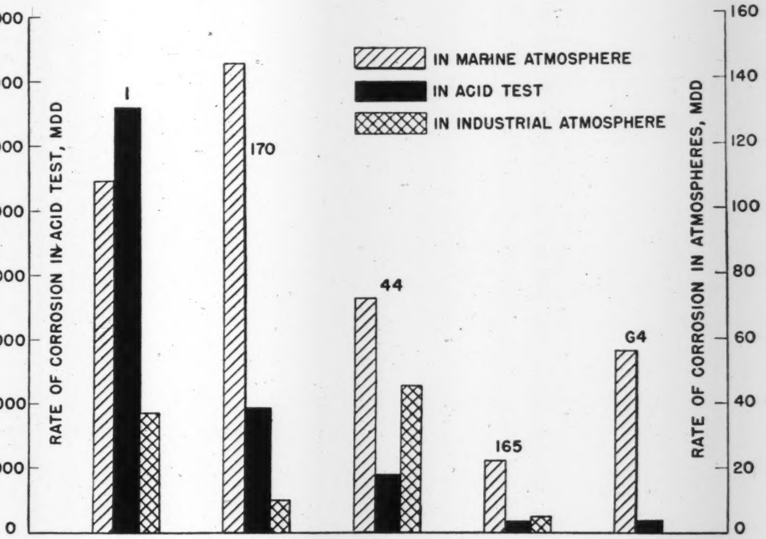

Fig. 1.—Comparison of Corrosion of Steels in Acid Tests and in Natural Atmospheres.

TABLE I.—CHEMICAL COMPOSITIONS OF STEELS COMPARED IN ACID TEST AND ATMOSPHERIC EXPOSURE.

Steel	Composition, per cent								
	Carbon	Manganese	Silicon	Sulfur	Phosphorus	Nickel	Copper	Chromium	Molybdenum
No. 1	0.085	0.43	0.005	0.051	0.10	0.002	0.01	0.02	...
No. 170	0.18	0.62	0.18	0.02	0.011	0.05	0.01	...	...
No. 44	0.016	0.016	0.002	0.029	0.005	<0.05	0.014	...	...
No. 165	0.17	0.66	0.17	0.02	0.033	0.23	0.21	...	...
No. G4	0.06	0.35	...	0.028	0.009	0.037	0.07	...	...

Even though the acid test for atmospheric corrosion resistance of steels has been discredited both by test and experience (10), it may be of interest here to provide further proof of its unreliability by citing some additional data.

Samples of a number of steels which demonstrated considerable differences in resisting atmospheric corrosion as disclosed by recent long-time tests under natural conditions of exposure have been subjected to the acid test, as described by Committee U in 1907. The results of these tests are compared in Fig. 1. The compositions of the steels are given in Table I.

It will be observed from Fig. 1 that the acid test failed to indicate the relative merits of the different steels in resisting atmospheric corrosion, and for the reason mentioned previously, this failure was most noticeable in the case of the high-phosphorus steel where the effect of phosphorus in accelerating attack in the acid test was just opposite to its effect in improving the protective qualities of the rusts formed under the natural conditions of atmospheric exposure, especially in the marine atmosphere.

This fundamental difference between the corrodibility of a steel, as measured by exposure to acids and its ability to form a protective rust under conditions of prolonged exposure to a natural atmosphere is responsible for the principal difficulty in devising an accelerated test that will afford a proper comparison amongst steels of different composition. The matter is complicated further by the fact that the relative merits of steels in marine atmospheres may not be the same as in industrial atmospheres, and, therefore, no accelerated test can be consistent with both kinds of atmospheric exposure.

OTHER ACCELERATED TESTS

Most of the accelerated tests that have been proposed tend to emphasize the corrodibility of the steel rather than the more important protective qualities of its rust. This criticism extends as well to the very elaborate testing machines such as have been developed both here (11) and in England (12) and which involve cycles of exposure to acid sprays, heating, drying, etc. While these devices are able to rate some steels in their proper order of merit in resisting corrosion by some atmospheres, they go astray when the steels do not exhibit a high degree of parallelism with respect to resistance to attack by the sprays used in the accelerated test machine and the protective qualities of the rusts they form in the natural atmospheres (13). While the corrosion-accelerating effect of sulfur and the protective effect of copper will be shown in both cases, the beneficial effects of nickel and chromium, and especially phosphorus, will be disclosed properly only by the natural tests.

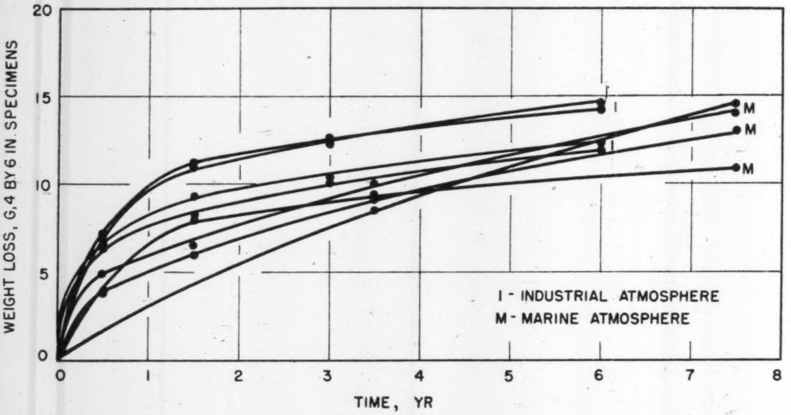

FIG. 2.—Typical Weight Loss *versus* Time Relationships in Industrial and Marine Atmospheric Corrosion Tests of Eight Low-Alloy Steels which Demonstrated Exceptionally Good Resistance to Corrosion.

An interesting attempt at a compromise between accelerated and natural tests was made some years ago by Pray and Gregg (14). These investigators allowed the steels to develop their usual rust films under conditions of natural exposure for a month. Then, instead of waiting for years of further exposure to the natural conditions to disclose the relative protective properties of the different rust films, they measured these qualities by exposing the rusted specimens to a much more aggressive environment in their laboratory.

While this procedure yielded more reliable results than did other methods of accelerated test which measured primarily the corrodibility of the steel itself, the Pray and Gregg method had the weaknesses that:

1. The abilities of the rust films to prevent attack by the aggressive laboratory environment did not necessarily parallel their abilities to provide protection against further atmospheric attack under the natural conditions under which the rust films were formed.
2. The rust films were not able to develop their full protective qualities during the time allowed for exposure under natural conditions.

With regard to item 2 above, the weight loss *versus* time data for a number of steels assembled in Fig. 2 indicate that for a standard 4 by 6-in. specimen exposed in the usual way at an angle of 30 deg from the horizontal facing south in an industrial atmosphere, about 11 g of iron must be converted into corrosion products before the full protective value of the rust has been achieved.

This observation is of particular significance in establishing a sort of minimum weight loss which it will be very difficult to reduce by the manipulation of the compositions of steels that rust.

It also suggests that the duration of atmospheric corrosion tests on specimens as described should be sufficiently long for the best steels to suffer weight loss to at least the extent indicated and need not be continued for more than a year or two after the limiting rate of further attack has been established by the formation of the rust films associated with the required extent of corrosion. In the case of industrial atmospheres, this critical time is about 4 yr as indicated by Fig. 2.

In marine atmospheres, the situation is somewhat different, as shown by the data in Fig. 2. Four steels exposed in a marine atmosphere and subject to corrosion to about the same extent as the four steels in the industrial atmosphere did not, with one exception, develop as protective rust films. Evidently more corrosion in a marine atmosphere is required before the full protective value of the rust is achieved, presumably because the basic sulfur compounds that confer protection in industrial atmospheres (8) are made more permeable by the development of soluble chlorides in marine atmospheres. This suggests that the opportunity for improvement in resistance to corrosion by a marine atmosphere is greater, though harder to achieve, than in an industrial atmosphere and that a longer time will be required to establish the relative merits of different compositions of the more corrosion-resistant steels, for example, 8 yr as compared with 4 yr for an industrial atmosphere.

RELATIONS BETWEEN RUST COLOR AND CORROSION

Many investigators of atmospheric corrosion have observed that there is a big difference in the color and texture of the rusts that form on different steels which exhibit differences in resistance to atmospheric corrosion. (This was observed in the tests undertaken by Committee A-5 (15) and the results of inspection of the test sheets included notes on the color and texture of the rusts.)

On the most severely corroded steels, the rusts tend to be very light in color and develop a very coarse texture. On the other hand, the more resistant steels form rust films that are of fine texture and are quite dark in color. The differences in color become apparent quite early in the exposure period and before the differences in texture are so evident.

Several years ago, the author had occasion to expose simultaneously a number of specimens of over a hundred steels of different compositions in the atmospheric test lot located about 800 ft from the ocean at Kure Beach, near Wilmington, N. C. It was observed that the rusts that developed on these several steels

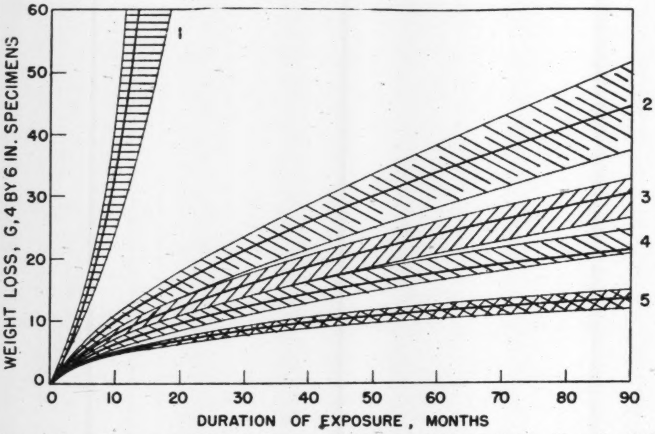

FIG. 3.—Relationship Between Weight Loss and Color of Rust Color Standard Steels in Marine Atmosphere at Kure Beach, N. C.

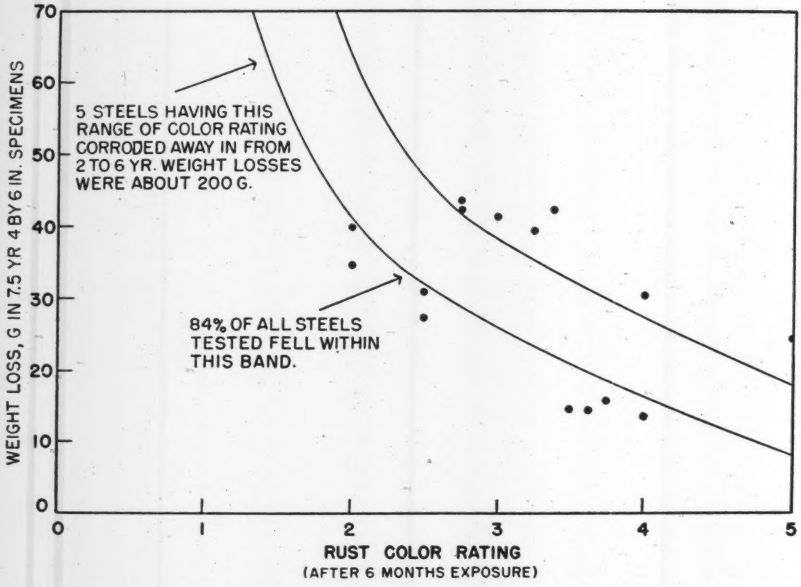

FIG. 4.—Relationship Between Rust Color Rating and Corrosion of Steels During 7.5 yr Exposure in Marine Atmosphere at Kure Beach, N. C.

exhibited a broad variation in color. To get some idea as to the relationship between these rust colors and the eventual corrosion of the several steels, selected standards of rust color were set up as represented by the rusts that formed on certain steels. The lightest color rust was given the number 1 and the darkest number 5. Number 2 was assigned to the rust a little darker than number 1 and number 4 to the rust a little lighter than number 5. The number 3 rust was intermediate in color between 2 and 4. Using these rust color standards for comparison, specimens of all the steels exposed were given rust color ratings after they had been exposed for 6 months. These ratings were made without the rater being aware of the identity of the specimens being rated.

TABLE II.—COMPOSITIONS OF STEELS USED FOR RUST COLOR STANDARDS.

Rust Color Rating of Steel After Exposure for 6 months	Composition, per cent								Estimated Ratio of Superiority[a] Over Copper Steel Rated No. 2
	Carbon	Manganese	Silicon	Sulfur	Phosphorus	Nickel	Copper	Chromium	
1.............	0.016	0.016	0.002	0.029	0.005	...	0.014	...	0.13
2.............	0.046	0.40	0.005	0.019	0.006	...	0.21	0.07	1.0
3.............	0.09	0.33	<0.01	0.034	0.065	0.40	0.22	0.19	2.0
4.............	0.11	0.43	0.18	0.031	0.012	1.52	1.09	...	2.1
5.............	0.17	0.58	0.26	0.013	0.007	4.98	0.09	...	4.0

[a] Based on weight loss *versus* time curves either when a steady rate had become established or after exposure for 7½ yr in a marine atmosphere.

The weight loss *versus* time curves for the steels selected as rust color standards are shown in Fig. 3 and illustrate the striking parallelism between the color of the rust and the resistance to atmospheric corrosion.

The relationship between these rust color ratings as they developed early in the test and the corrosion resistance of the steels as measured by long time exposure is shown in Fig. 4.

The over-all consistency of the relationship between the color of the rust and the corrosion of the steel is remarkable—especially if allowance is made for the fact that if the phosphorus content of a steel is over about 0.08 per cent or the chromium content is over 2 per cent it will perform as though its rust color were about 1 number higher (darker) than it appears. Figure 4 has been plotted with these adjustments for phosphorus and chromium made.

It would appear, then, that the prediction of ultimate and relative performance over a long period of exposure may be based on the rating of the color of the rust that forms on a steel early in its exposure. The degree of reliability will compare very favorably with the results of "accelerated" tests of the types that have been proposed and used from time to time. The assignment of these rust color numbers to any unknown steels to be rated will be facilitated by the exposure at the same time of specimens of steels representing the original rust color standards as described here.

In applying rust color ratings to predictions of performance, the figures given in Table II will be helpful.

Effects of Position in Test

So as to facilitate comparison of results secured at different times and places, the Society, as well as many independent investigators of atmospheric corrosion in this country, has standardized on the exposure of steel corrosion test specimens at an angle of 30 deg from the horizontal with the racks facing south, as illustrated in Fig. 5.

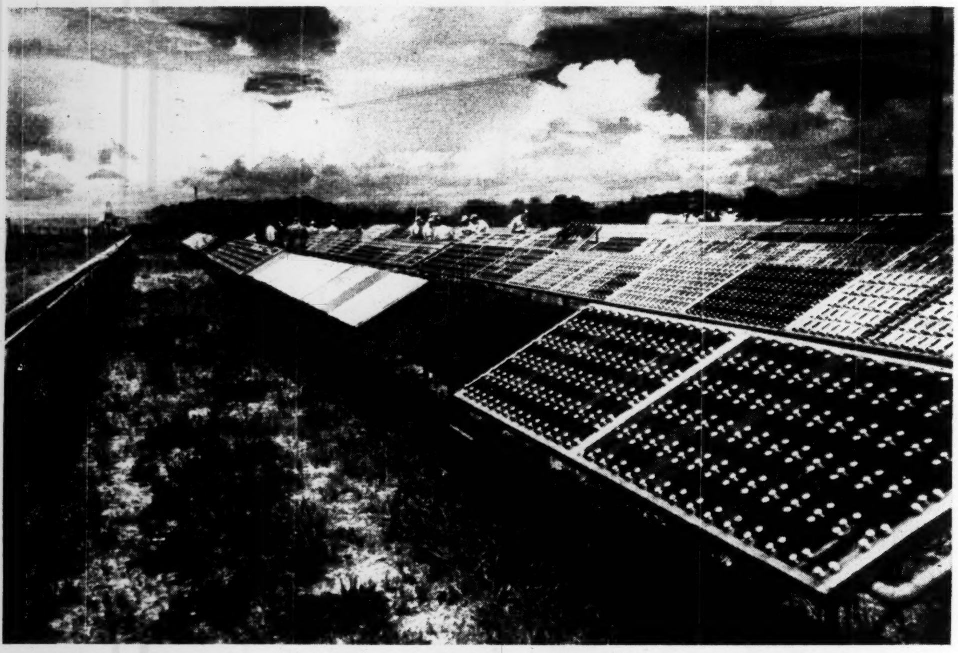

FIG. 5.—Exposure of Specimens on Atmospheric Test Racks at Kure Beach, N. C.

This method of exposing bare test specimens influences the results secured to the extent that it permits washing of corrosive compounds from the upper surfaces by heavy rains and frequent thorough drying of the rust films by the sun so as to enhance their protective value by the mechanisms described by Copson.

The groundward surfaces of specimens do not benefit from these washing and drying effects to the same extent, and, therefore, the rust films do not become as protective. H. S. Rawdon (16) reported observations to this effect, and C. P. Larrabee (17) established that in an industrial atmosphere about 60 per cent of the corrosion occurs on the groundward surfaces and 40 per cent on the skyward surfaces of specimens exposed as described.

It is the common practice of British investigators (18) to expose atmospheric test specimens vertically. Tests on steel specimens exposed in this way as compared with ASTM practice have been reported by E. S. Taylerson (19). Some of his results, plus some data from tests by the author in the marine atmosphere at Kure Beach, N. C., are shown in Table III. Apparently, specimens exposed vertically do not develop as protective rusts as those exposed at an

TABLE III.—EFFECT OF POSITION OF SPECIMENS ON ATMOSPHERIC CORROSION IN 1 YR TEST.

Test Location	Ratio of Corrosion of Specimens Exposed Vertically to That of Specimens Exposed 30 deg from the Horizontal
Kearny, N. J.	1.25
Vandergrift, Pa.	1.26
South Bend, Pa.	1.20
Kure Beach, N. C.—800 ft from ocean	1.25
Kure Beach, N. C.—80 ft from ocean	1.41

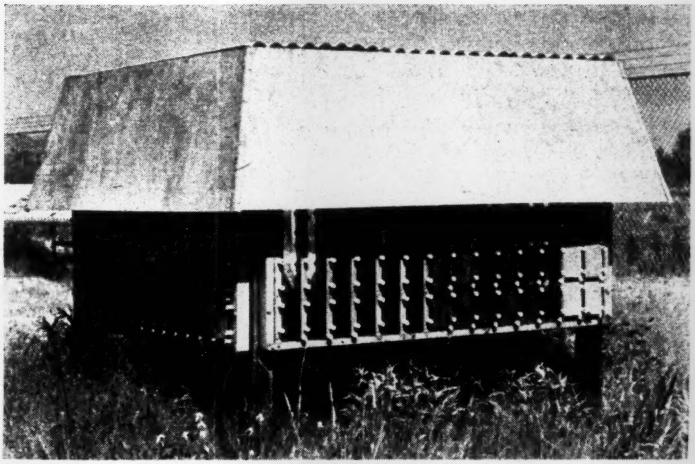

FIG. 6.—Structure Designed by C. P. Larrabee to Study Effects of Partial Shelter on Atmospheric Corrosion of Steel.

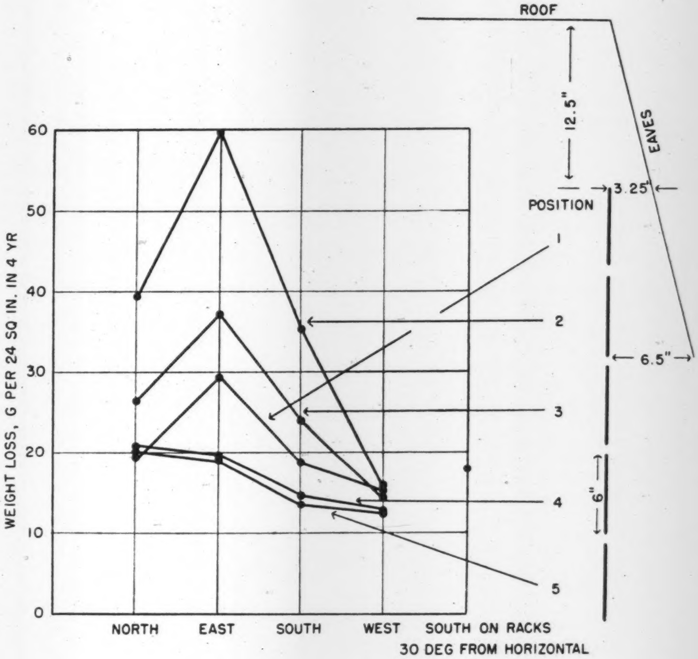

FIG. 7.—Effect of Degree of Shelter and Orientation on Corrosion of Carbon Steel (0.04 Cu) Specimens Exposed at Kure Beach, N. C.

angle, especially in the very severe environment close to the ocean. The vertical exposure has the disadvantage that a few degrees variation from the specified 90 deg will have a much greater effect on the extent to which the surfaces will be shaded than will a similar variation from a specified 30-deg position.

In the extensive atmospheric corrosion test program instituted in Sweden in 1938 large specimens have been used so as to permit exposure of part of the surfaces horizontally, part vertically, and part at 45 deg (20).

A striking demonstration of the effects of shelter has been provided by some tests carried out by C. P. Larrabee of the Research and Development Laboratory, United States Steel Co., at the test site 800 ft from the ocean at Kure Beach, N. C. These are to be described in detail in an as yet unpublished paper by Mr. Larrabee who has kindly permitted the present author to use some of his data to illustrate the point under discussion.

Specimens of steel were arranged in racks which formed the sides of the structure shown in Fig. 6. The structure was located so that its sides faced north, south, east, and west. Each vertical row of specimens was made up of 5 pieces of the same steel with the inward surfaces covered with a protective coating so as to confine corrosion to the outward surfaces. The extent to which the exaggerated eaves shaded and sheltered the specimens is indicated by the diagram in Fig. 7.

The overhanging eaves sheltered the upper specimens against the destructive effects of wind blown salt, as well as against the protective effects of the sun and rain so that, as will be seen from the data plotted in Fig. 7, the most vulnerable specimen was that in position 2 facing east which was far enough under the eaves to miss the beneficial effects of the rain and sun, but not far enough to escape the corrosive effect of wind borne salt. (Based on data provided by A. H. Woodcock (21) H. R. Copson has calculated that a 4 by 6-in. specimen might come into contact with as much as 170 g of salt deposited from the atmosphere near the ocean in a year.)

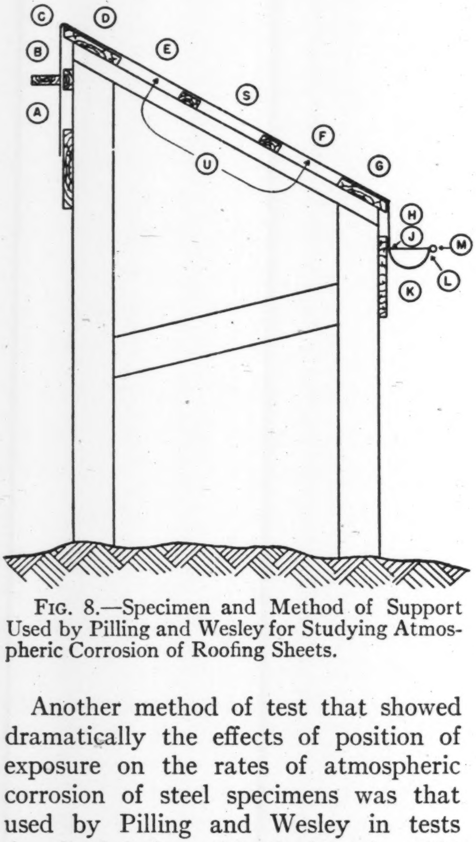

FIG. 8.—Specimen and Method of Support Used by Pilling and Wesley for Studying Atmospheric Corrosion of Roofing Sheets.

Another method of test that showed dramatically the effects of position of exposure on the rates of atmospheric corrosion of steel specimens was that used by Pilling and Wesley in tests described before this Society in 1940 (22), and illustrated by Fig. 8. The progress of failure of specimens of this sort was in the order shown in Table IV with reference to the sections identified in Fig. 8.

The advantage of an exposure that permits rust films to be rinsed regularly and dried frequently is clearly indicated by the results of this test.

Results such as have been just described show that tests on specimens exposed on racks in the usual ways measure the corrosion resistance of steels under somewhat favorable circumstances as compared, for example, with exposure or use under conditions of partial shelter where rust films would not have equal opportunity to dry thoroughly and thus develop their best protective qualities. Thus, the advantages of one steel over another demonstrated by exposure tests of this sort may not be realized fully in all practical applications involving resistance to atmospheric corrosion. At the same time, the order of merit is not disturbed by the change in exposure conditions, and the alloy steels found best by the conventional atmospheric tests never appear at a disadvantage as compared with unalloyed steels even under unfavorable conditions of use involving atmospheric corrosion. Furthermore, as pointed out by Copson (23), the extent of pitting of sheltered specimens is no greater than that of those exposed boldly so that where resistance to perforation is a prime consideration the data secured by bold exposure will be applicable to sheltered conditions as well.

TABLE IV.—ORDER OF FAILURE OF OPEN-HEARTH COPPER STEEL IN SIMULATED ROOFING SHEET TEST IN INDUSTRIAL ATMOSPHERE.

Section of Specimen[a]	Life in Months
K	25
J and L	25
A and B	130
C to G	178

[a] As shown in Fig. 8.

These observations can also serve as a guide in the design and fabrication of structures and equipment so as to get full benefit from the superior corrosion resistance of alloy steels which reaches a maximum when there is a chance for rust films to dry out frequently. When this opportunity cannot be arranged, then it will be desirable to provide supplementary protection to the surfaces that will require it—as by painting the lapped surfaces of roofing and siding sheets. By this means, the durability of these critical areas can be increased so as to be able to take full advantage of the extra corrosion resistance of the freely exposed surfaces.

Somewhat along the same line are applications where protective rust films are disturbed regularly by abrasion, as in the case of the interior surfaces of railroad hopper cars. In such circumstances, the over-all problem is complicated by abrasion of the metal that accompanies removal of the rust films and by the compensating fact that many of the steels that demonstrate superior resistance to atmospheric corrosion when undisturbed by abrasion also provide extra resistance to the abrasion that prevents full demonstration of their superior corrosion resistance. The net result is that the over-all advantage of the alloy steels is maintained even though the extent of this advantage will vary from circumstance to circumstance in line with the differences in resistance to abrasion and corrosion that exist and the relative importance of these factors in determining over-all life.

This sort of situation and the interplay of the forces of deterioration and the resistances to these forces were illustrated very well by the service records of different kinds of steel in hopper car service as reported recently by B. J. Kelly (24). Mr. Kelly's paper was based on tests of railroad hopper cars in coke hauling and coal hauling service. In the former, abrasion is considered to be most important, and in the latter, corrosion. Furthermore, different sections of the cars are subjected to abrasion and corrosion to different degrees—in cross ridge sheets resistance to abrasion is most important, while in side and end sheets improved resistance to atmospheric corrosion leads to longer life.

In the tests mentioned, cars were constructed using one steel for one half of a car and another steel for the other half. In this way three steels were compared:

Steel A—copper steel.

Steel B—An abrasion-resisting steel having the atmospheric corrosion resistance of copper steel.

Steel C—A low-alloy high strength steel having the abrasion resistance of steel B and about twice the atmospheric corrosion resistance of copper steel A.

Examination of sheets removed from the test cars after service for several years indicated the relative merits of the different steels as summarized in Table V.

TABLE V.—RELATIVE MERITS OF DIFFERENT STEELS IN RESISTING CORROSION AND ABRASION IN RAILROAD HOPPER CARS.

Service	Advantage over Copper Steel A			
	In Cross Ridge Sheets		In Intermediate Side Sheets	
	Abrasion-Resisting Steel B	Abrasion- and Corrosion-Resisting Steel C	Abrasion-Resisting Steel B	Abrasion- and Corrosion-Resisting Steel C
Coke	1.1	1.4	1.3	2.4
Coal	1.0	2.0	1.4	4.3

It will be noted in comparing the relative merits of the different steels in the same parts, the advantage of superior corrosion resistance was realized to the greatest extent in the side sheets where corrosion was more of a factor than abrasion. Similarly, in comparing coke service, where abrasion was a major factor, with coal service where corrosion was predominant, the corrosion-resisting steel showed the greatest advantage in the more corrosive service. And, in spite of the complicating effects of abrasion, the steel that demonstrated extra resistance to corrosion in conventional atmospheric tests also performed best in hopper car service.

The dependence of different steels on their rust film characteristics and the opportunity for rust films to become dry in order to develop their most protective qualities will account, of course, for the failure of steels to maintain the advantage demonstrated by bold exposure to the atmosphere when they are exposed or used under conditions where there is no opportunity to form similar rust films—as under conditions of immersion in waters or other corrosive liquids. This point was illustrated by the results of the ASTM tests in fresh water and mine waters carried out in parallel with the atmospheric exposures of the several kinds of steels and irons and discussed along with these tests by Taylerson and Kendall (25) in their paper in which they analyzed the results of this test program.

The several varieties and compositions of steel and iron were exposed to corrosion by Washington, D. C., tap water, Severn River water and Calumet Mine water. By statistical analysis of the results, these authors concluded that "variation of the common elements, either singly or in combination, within the range of composition and under the conditions of these tests, has no material influence on corrosion resistance and is entirely secondary to external conditions." This conclusion has been supported by several tests by the present author which have involved exposure of many kinds of steel and iron to corrosion by natural waters.

However, when immersed in particular corrosive media, steels may differ greatly in resistance to corrosion. This difference may be due to some specific effect of a constituent in improving inherent resistance to attack as in the case of copper steels in sulfuric acid and nickel steels in strong alkalies, or in impairing resistance to corrosion as in the case of high phosphorus content steels in acid solutions. In additon, certain steels may be able to develop more protective corrosion product films than others in particular environments, as well as in the atmosphere. For example, it seems likely that the superior corrosion resistance of steels containing nickel or chromium in the peculiar corrosive media (principally carbonic acid) encountered in gas condensate wells as illustrated in Fig. 9 is due primarily to the formation of unusually protective corrosion product films (26).

The principle to be followed is that it is not safe to assume that effects of composition on the corrosion resistance of steels will pertain to all corrosive environments. Relative resistance to attack as demonstrated, for example, by atmospheric corrosion tests may not be maintained in other corrosive media, especially under conditions of submersion. This does not mean that important differences in corrosion resistance may not exist but suggests that each corrosive environment must be treated as a special case in appraising the relative merits of different kinds of iron and steel, as well as other metals and alloys.

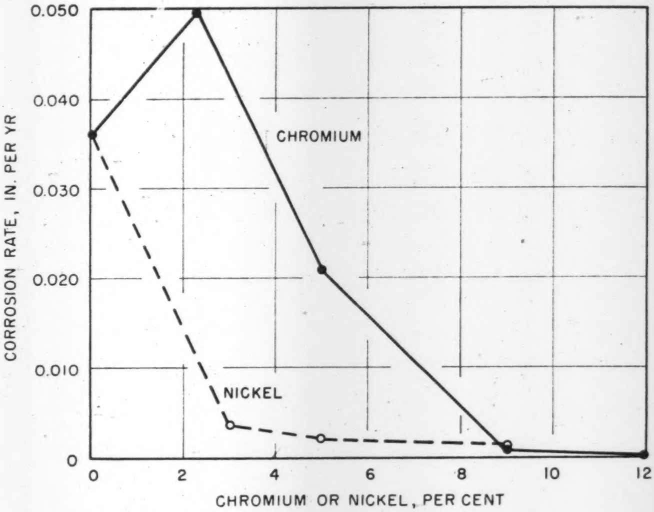

FIG. 9.—Effects of Chromium and Nickel on Resistance of Steels to Corrosion in Condensate Gas Well.

LONG-TIME TESTS OF DIFFERENT STEELS AND IRONS

As mentioned previously, one of the major testing activities of the Society has been the establishment of the differences in performance between different kinds of iron and steel when exposed under identical conditions in different atmospheres. The principal program involved the exposure of large corrugated sheets at three locations—Pittsburgh, Fort Sheridan, and Annapolis, under the auspices of Committee A-5 (15). These tests were started in 1916 and have gone to completion at the test sites except Annapolis where specimens remain in test. It is unnecessary here to review the results of these tests which can be found in the several reports of Committee A-5. However, particular reference should be made to the excellent analysis of some of the early and most significant results by E. S. Taylerson and V. V. Kendall (25). Their principal findings from the data may be summarized as follows:

In the sulfurous industrial atmosphere at Pittsburgh, copper, and especially a combination of copper and phosphorus, had a strongly beneficial effect on corrosion resistance. Their influence was greater in the steels than in the irons.

In the rural atmosphere, the attack was much less severe, but, in general, the indications from the Pittsburgh tests applied here as well.

The Annapolis atmosphere was supposed to represent a sea coast one, but in this respect it must be characterized as extremely mild and probably more like a rural atmosphere than a sea coast one. The tests at this location are still in progress and the results being secured do not disturb the general conclusions reached from the tests in the more aggressive environments.

The method of inspection and the criterion of failure, visible perforation of the test sheets more than $\frac{1}{4}$ in. from an edge left a good deal to be desired, since it has been observed in many cases that practically complete destruction of a specimen may occur before any perforation is visible through the heavy layers of rust that form. This is illustrated by Fig. 10 which shows on the left a rusted specimen of steel as it was taken from a test rack without any visible evidence of perforation and on the right the complete destruction of the steel when the stratified layers of compact rust were broken to show that no unattacked steel remained. This specimen measured only 4 by 6 in. With much larger specimens, such as used in some of the A-5 tests, the development of visible perforations would be favored by the greater vibration of the large sheets so as to uncover perforations by shedding the rust; but even with the large specimens, many perforations may remain undetected.[5]

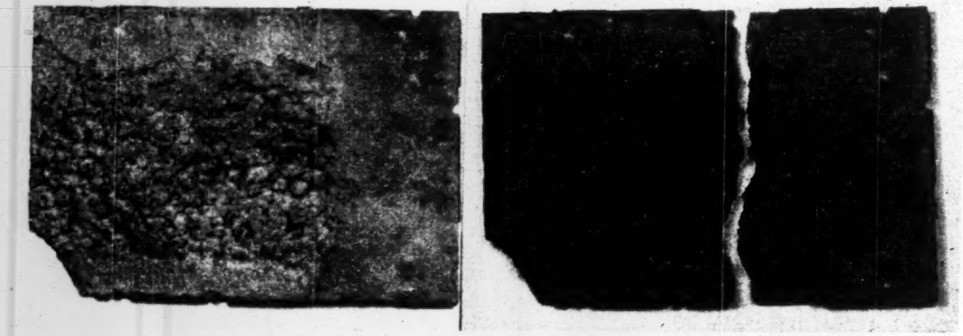
As Taken From Rack Same Specimen Broken

FIG. 10.—Specimen of Steel Corroded to Destruction Without Perforations at Kure Beach, N. C.

Other shortcomings of the use of time to visible perforation as the criterion of corrosion resistance are:

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[5] The discovery of perforations in the sheets from the Fort Sheridan exposure that had not been observed when the sheets were inspected on the racks but which appeared when they were cleaned in acid for weight loss determinations was noted in the 1946 report of Committee A-5. There was a further statement that negligible corrosion had occurred during the period of storage under favorable conditions between when the specimens were taken from the racks and when they were cleaned to remove corrosion products. These holes, therefore, were present when the sheets were inspected, but were undetected because they were covered with rust.

1. The removal of rust films to facilitate inspection for perforation prior to termination of the exposure will change the natural performance of the steels and is, therefore, not tolerable.

2. The recording of a perforation establishes only the time to failure and provides no idea of the progress of corrosion up to the point of failure.

TABLE VI.—COMPARATIVE BEHAVIOR OF DIFFERENT STEELS IN TERMS OF WEIGHT LOSS AND PITTING WHEN EXPOSED FOR 3½ YR IN MARINE ATMOSPHERE 80 FT FROM OCEAN AT KURE BEACH, N. C.

Steel	Composition, per cent								Advantage Over Steel No. 165[a] With Respect To	
	Copper	Manganese	Silicon	Phosphorus	Nickel	Copper	Chromium	Molybdenum	Weight Loss	Pitting
No. 165	0.17	0.66	0.17	0.033	0.23	0.21	...	...	...	...
No. 166	0.15	0.64	0.17	0.12	0.22	0.24	...	...	1.9	1.0
No. 168	0.09	0.58	0.15	0.035	0.69	0.26	0.29	0.15	2.9	1.4
No. 169	0.14	0.24	0.10	0.007	4.91	0.02	...	...	5.8	6.7

[a] Steel No. 165—rate of weight loss, 29 mg per sq dm per day; maximum pitting, 0.08 in.

3. The time to perforation may be influenced considerably by the random occurrence of pits that happen to meet after starting from opposite sides of a sheet. This chance meeting of pits may be determined only slightly by the composition of the steel and, therefore, will interfere with observations of effects of steel composition.

Although, for the reasons cited, the scatter in results of tests based on time to perforation of specimens is likely to be large, proper conclusions may be reached if the number and size of specimens are great enough. This, fortunately, was the case with the large-scale tests undertaken by Committee A-5, the results of which were subjected to statistical analysis by E. S. Taylerson and V. V. Kendall (25) as discussed previously.

However, for the more accurate measurement of corrosion resistance and differences amongst materials, the author prefers the exposure of specimens small enough to be weighed accurately after careful removal of corrosion products. A sufficient number of specimens should be exposed initially to permit their withdrawal from test in appropriate groups after at least three time intervals. In a particular program of this sort, 16 specimens of each kind were exposed, to be withdrawn from test for re-weighing as follows:

2 after ½ yr
2 after 1½ yr
4 after 3½ yr
4 after 7½ yr
4 after 15 yr

If this program were to be repeated, a better procedure would be to remove the specimens as follows:

4 after 1 yr
4 after 3 yr
4 after 7 yr
4 after 15 yr

Actually, the 15-yr specimens could be dispensed with since the relative merits of the steels will become established in 7 yr, and any longer exposure would serve only to confirm the merit of the best steels. The ½-yr exposure can be eliminated because such early results do not have much practical significance.

It is good practice to determine the depth of pitting, as well as weight loss, in examining specimens after exposure. This may not change the picture much so far as the relative merits of the worst and best steels are concerned but may have an important bearing on distinctions that may be made between good steels that show similar losses in weight (23) as illustrated by the data in Table VI.

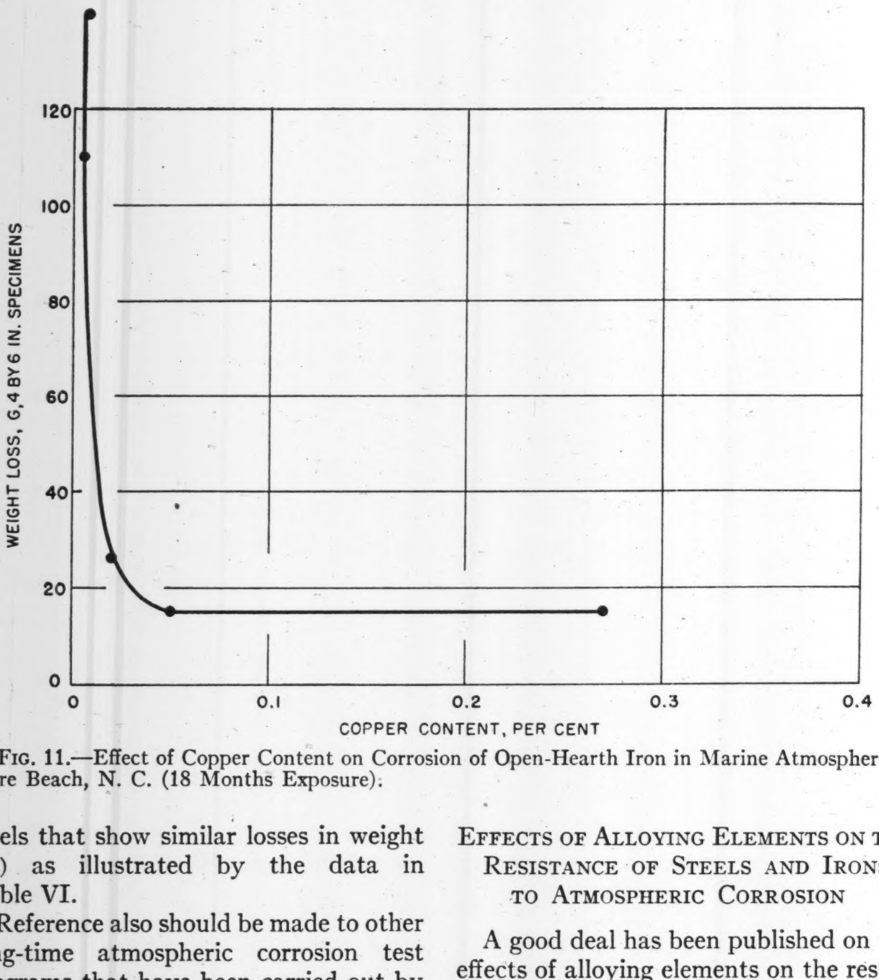

FIG. 11.—Effect of Copper Content on Corrosion of Open-Hearth Iron in Marine Atmosphere at Kure Beach, N. C. (18 Months Exposure).

Reference also should be made to other long-time atmospheric corrosion test programs that have been carried out by Committee A-5. These include the large-scale tests of zinc-coated and bare steels and of line hardware that are still in progress as described in the numerous reports of this committee (27). In addition, are the very extensive tests of coated and bare wires and fencing being undertaken by Subcommittee XV of A-5 which also are described in the reports of this committee. These tests have been well planned and executed and are yielding interesting results of great practical value.

EFFECTS OF ALLOYING ELEMENTS ON THE RESISTANCE OF STEELS AND IRONS TO ATMOSPHERIC CORROSION

A good deal has been published on the effects of alloying elements on the resistance of steels to atmospheric corrosion (28). The work by Committee A-5 on copper steels has already been mentioned. Considerable additional data have been summarized by Hudson (29), Larrabee (30), and by Pilling and Wesley (22). More attention has been given to behavior in rural and industrial atmospheres than in strictly marine atmospheres. Therefore to add to this picture

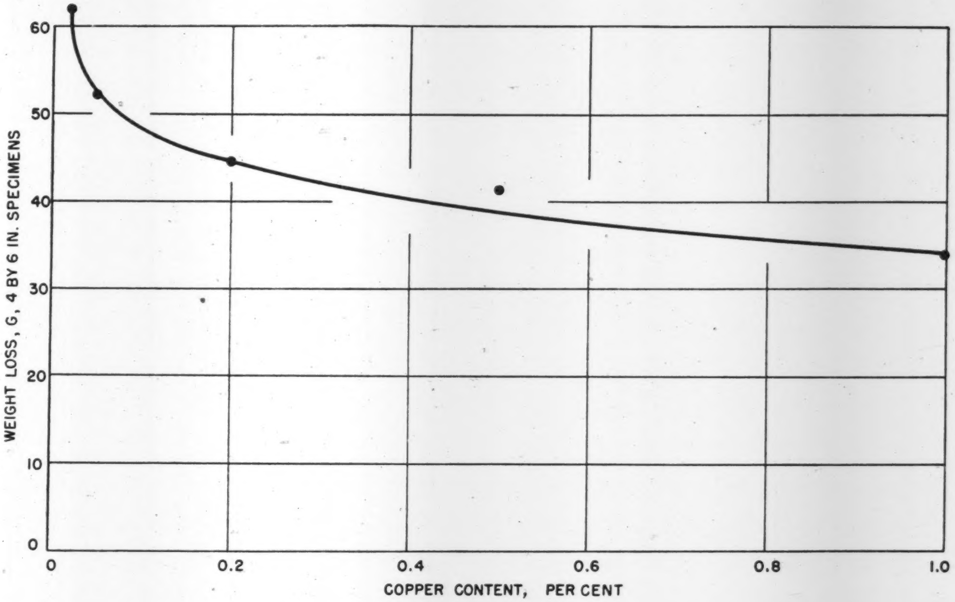

Fig. 12.—Effect of Copper Content on Corrosion of Open-Hearth Steel in Marine Atmosphere a Kure Beach, N. C. (90 Months Exposure).

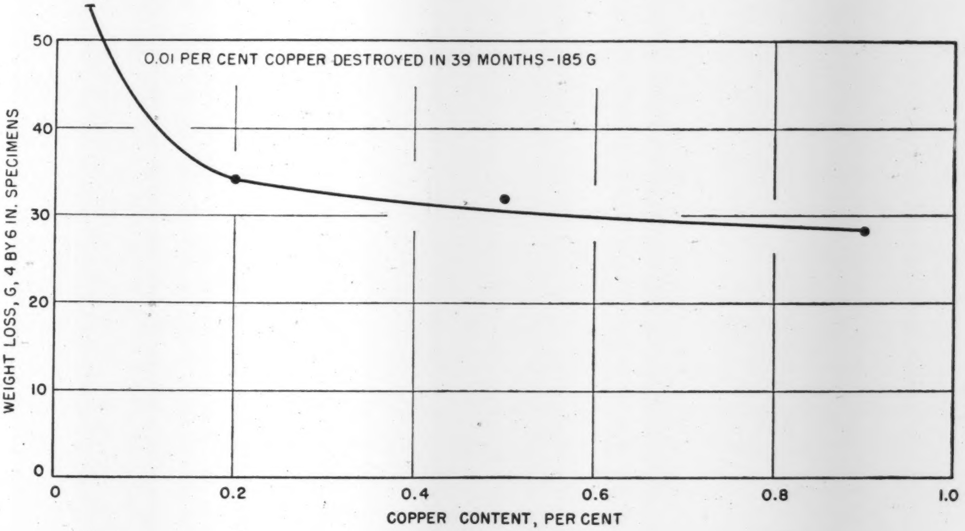

Fig. 13.—Effect of Copper Content on Corrosion of Bessemer Steel in Marine Atmosphere at Kure Beach, N. C. (90 Months Exposure).

there will be presented here some results of tests which have shown the separate effects of copper, nickel, and chromium on the behavior of steels and irons in the marine atmosphere 800 ft from the ocean at Kure Beach, N. C. All specimens used in these investigations measured 4 by 6 in. and were exposed in the standard way at an angle of 30 deg from the horizontal facing south. All specimens were pickled to remove mill scale and rust prior to exposure.

In order to avoid any peculiar effects of position on the racks, the specimens were distributed at random in accordance with instructions provided by Wm. Campbell of the Bell Telephone Laboratories. Sixteen specimens of each composition were exposed and removed as follows: 2 after six months, 2 after 18 months, 4 after 42 months and 4 after 90 months. The balance of the surviving specimens are scheduled for removal after $15\frac{1}{2}$ yr.

The specimens removed after the first three test periods were cleaned electrolytically as cathodes in inhibited dilute sulfuric acid. The last set of specimens was cleaned by the sodium hydride descaling process through the courtesy of Mr. O. B. Ellis of the Armco Steel Corp.

Loss in weight during exposure was used to measure corrosion.

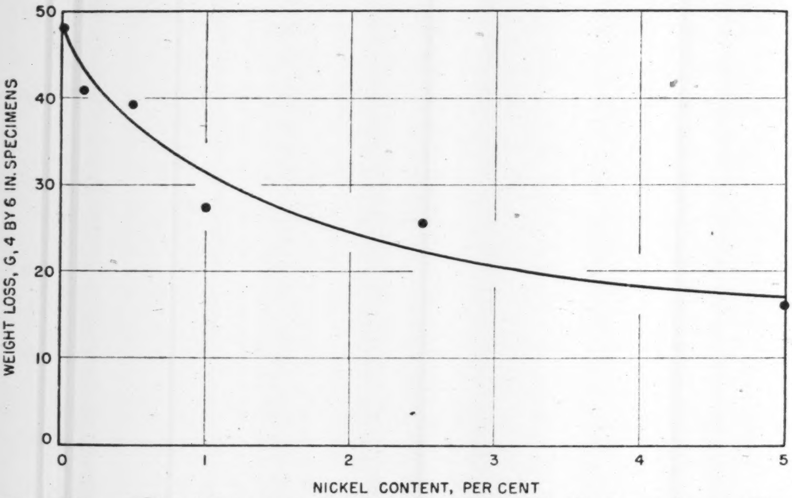

Fig. 14.—Effect of Nickel Content on Corrosion of Steel in Marine Atmosphere at Kure Beach, N. C. (90 Months Exposure).

Effects of Copper:

The studies of the effects of copper were divided into three parts: copper in open-hearth irons, copper in open-hearth steels and copper in bessemer steels.

The effects of very small amounts of copper in the open-hearth irons as shown in Fig. 11 were very much the same as discovered by D. M. Buck (31) in his classical studies of the effects of copper in steels in an industrial atmosphere.

It will be observed from the data in Fig. 11 for the open-hearth irons that in the marine atmosphere as well as in an industrial atmosphere most of the effect due to the presence of copper is achieved with the first 0.03 per cent or so added. There can be as much as a ten-fold difference in corrosion between irons carrying 0.005 per cent and 0.05 per cent copper.

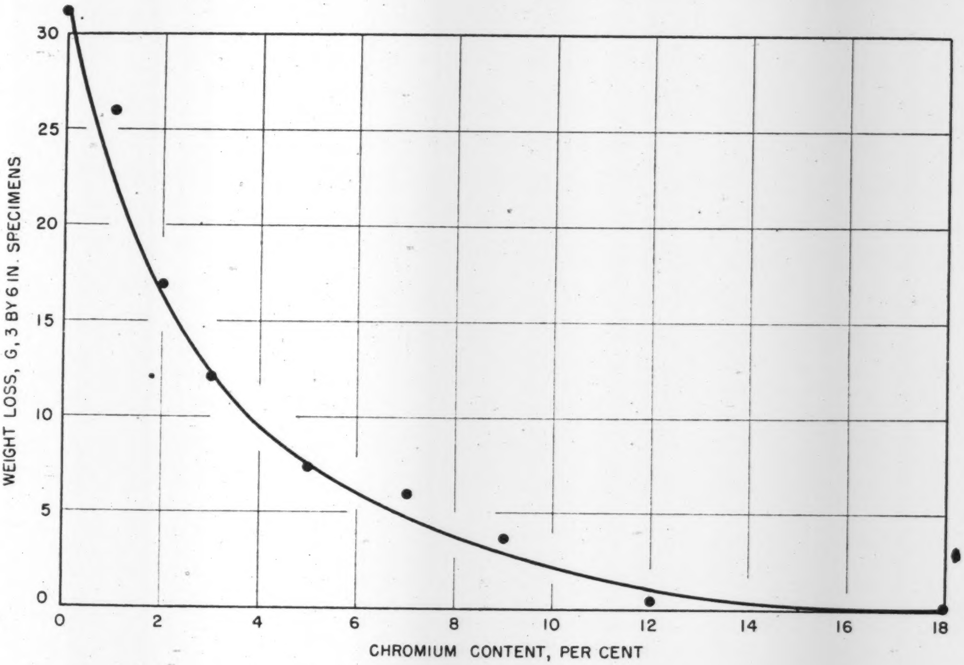

Fig. 15.—Effect of Chromium Content on Corrosion of Steel in Marine Atmosphere at Kure Beach, N. C. (90 Months Exposure).

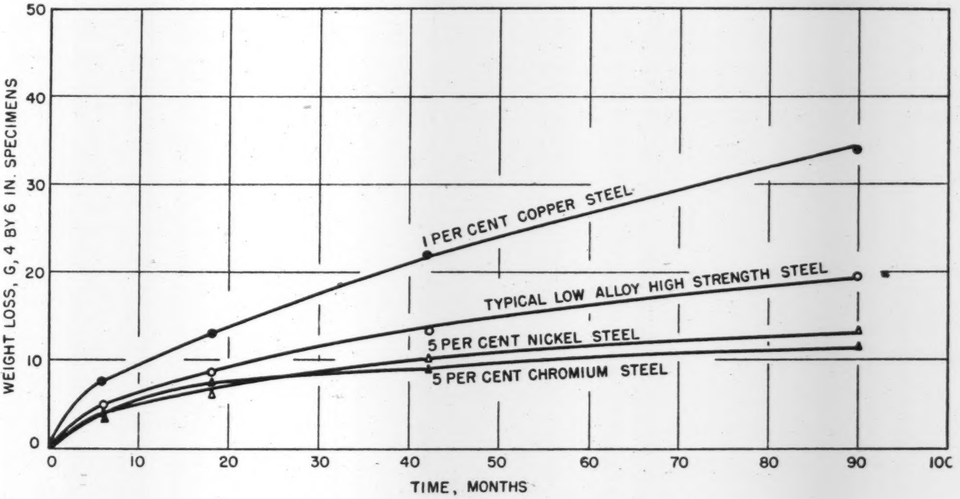

Fig. 16.—Weight Loss *versus* Time Curves for Steels in Marine Atmosphere at Kure Beach, N. C.

Effects of larger amounts of copper in open-hearth and bessemer steels are shown by the data plotted in Figs. 12 and 13. The contribution of phosphorus to corrosion resistance is indicated by the lower weight losses of the bessemer copper steels.

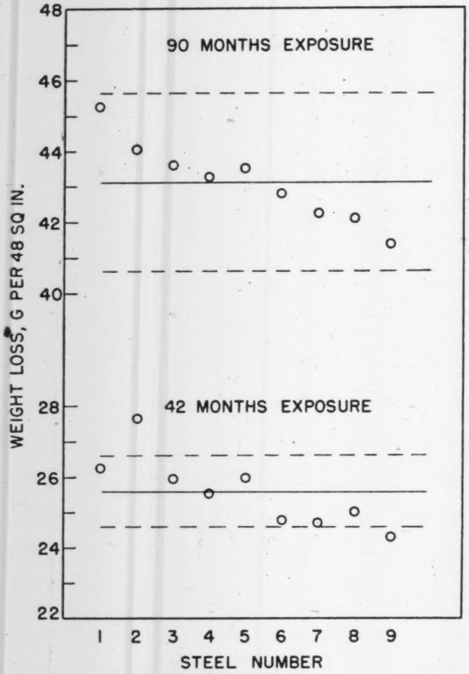

FIG. 17.—Control Charts in Statistical Analysis of Data on Atmospheric Corrosion of Copper Steels.

The great effect of copper in concentrations ordinarily considered as residual serves to make carbon steel of undefined copper content an unsatisfactory basis of comparison in describing the atmospheric corrosion resistance of other steels. A much more satisfactory standard is the ordinary copper steel which contains about 0.20 per cent copper.

Effects of Nickel and Chromium:

Similar data with respect to nickel are shown in Fig. 14 and for chromium in Fig. 15. Here, the base line steels with no added nickel or chromium contained about 0.04 per cent copper.

The weight loss *versus* time curves for 1 per cent copper, 5 per cent nickel, and 5 per cent chromium steels are plotted in Fig. 16 which also includes a line representative of the performance of the proprietary, complex, low-alloy high-strength steels being manufactured by several producers.

Amongst the several commercial steels included in this program were specimens of copper steel from nine commercial melts supplied by different producers.

TABLE VII.—COMPOSITIONS OF COMMERCIAL OPEN-HEARTH COPPER STEELS INCLUDED IN MARINE ATMOSPHERIC CORROSION TESTS AT KURE BEACH, N. C.

Steel	Composition, per cent						
	Carbon	Manganese	Phosphorus	Sulfur	Silicon	Copper	Nickel
No. 1....	0.06	0.26	0.007	0.032	0.01	0.19	0.06
No. 2....	0.05	0.40	0.006	0.019	0.005	0.21	...
No. 3....	0.08	0.34	0.008	0.023	0.01	0.22	...
No. 4....	0.06	0.32	0.010	0.023	0.01	0.24	...
No. 5....	0.03	0.17	0.006	0.026	0.01	0.27	...
No. 6....	0.026	0.24	0.009	0.035	0.004	0.27	...
No. 7....	0.05	0.31	0.008	0.018	0.01	0.27	...
No. 8....	0.04	0.31	0.004	0.021	0.01	0.30	0.05
No. 9....	0.08	0.42	0.010	0.023	0.01	0.30	...
Range...	0.03 to 0.08	0.17 to 0.42	0.004 to 0.010	0.018 to 0.035	0.005 to 0.010	0.19 to 0.30	...

The results of the tests on these steels were used as the basis for a statistical analysis of the data to illustrate this method of treating data as prescribed by the Society in its Manual on Presentation of Data.

The results of this analysis are shown graphically in Fig. 17 for the compositions described in Table VII.[6] This shows excellent agreement amongst the duplicate specimens of each steel and also remarkable consistency amongst the several lots of copper steel from the different sources. Outside of a slight trend towards lower weight loss as the copper content of the steels increased, the data indicated that the other elements present in the steels had no significant effect within the ranges of concentration in which they were present.

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[6] Some details of this statistical analysis by E. A. Tice are given in the Appendix, see p. 578.

In general, the statistical analysis of this particular set of data demonstrates the reproducibility of data from tests of this sort and lends confidence to the interpretation and application of data based on losses in weight of atmospheric corrosion test specimens.

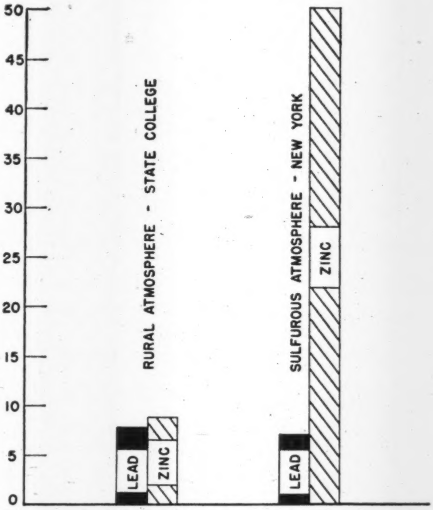

FIG. 18.—Effect of Sulfurous Compounds in Accelerating Corrosion of Zinc and Protecting Lead in the Atmosphere.

Those interested in a particularly fine example of the application of statistical methods to the interpretation of data from a complex test with many variables in the details of the test as well as in the compositions of materials tested are referred to the report of Committee TP-1 (26) of the National Association of Corrosion Engineers. The report includes a statistical treatment of the data by V. V. Kendall.

The importance of statistical methods in corrosion tests has been emphasized also by Kendall and Taylerson (25), Mears, Walton, and Eldridge (32), Passano (33), and in the reports of Subcommittee VI of Committee B-3 (34).

COMPARISON OF ATMOSPHERES

Unfortunately, we still do not have any convenient way to characterize the atmosphere in any particular locality as to its corrosive characteristics. We are not even sure which properties of an atmosphere or climate are most significant from the corrosion standpoint. We are as yet unable to list and measure factors of the weather in a way that will permit us to relate them systematically to corrosion that has been observed or to predict precisely the performance of materials on the basis of their behavior somewhere else—as at one of the established atmospheric corrosion testing stations.

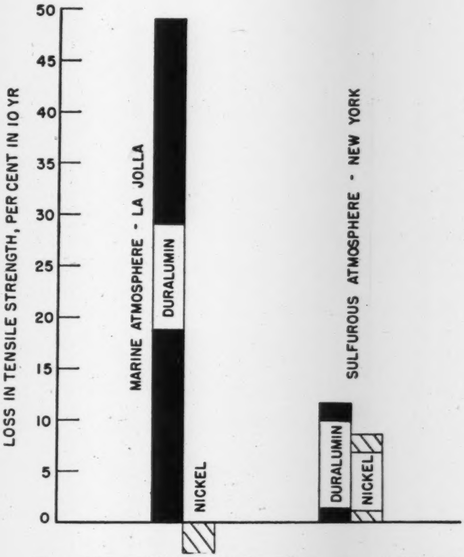

FIG. 19.—Differences in Response of Nickel and Duralumin to Corrosive Effects of Different Atmospheres.

Subcommittee VII of Committee B-3, Weather Factors in Corrosion, has been giving this subject some attention. It may also benefit from the work of the newly formed Committee D-22 on Methods of Atmospheric Sampling and Analysis.

The root of the difficulty is the fact that all materials do not respond alike to the factors that influence atmospheric corrosion. Certain contaminants of the atmosphere aggravate corrosion of some metals, yet tend to protect others, as in the case of the response of zinc and lead respectively to the presence of sulfuric acid in the atmosphere. An idea of the reactions of different metals to different climatic conditions can be obtained from the results of the extensive atmospheric corrosion tests carried out by Subcommittee VI of Committee B-3. See especially the report in volume 44 of the *Proceedings* and analyses of the data in the Symposium on Atmospheric Exposure Tests on Non-Ferrous Metals published by the Society in 1946.

The points under discussion are illustrated by the data from the Subcommittee VI tests plotted in Fig. 18 which provides the comparison between zinc and lead in industrial and rural atmospheres, and Fig. 19 which provides a comparison between nickel and an aluminum alloy in a marine and industrial atmosphere. The latter data disclose that while both atmospheres are destructive to this aluminum alloy,[7] only the industrial atmosphere has an appreciable corrosive effect on nickel.

Such results have a bearing on the common use of the corrosion of iron to measure the corrosivity of an atmosphere. While the corrosion of iron will disclose the potential corrosivity of an atmosphere towards other metals, it will not establish that any other metal will be corroded significantly in the atmospheres most corrosive to iron. Similarly, the same relation between corrosivity of different atmospheres towards iron will not necessarily extend to a number of metals and alloys.

A practical feature from the standpoint of describing weather characteristics is the difficulty of making accurate measurements of and distinctions between rain, dew, fog, and humidity. With regard to the latter factor, the humidity at which one metal will corrode is different from that for another and is influenced also by the hygroscopic properties of certain products of corrosion, as well as by particles of dust that aggravate atmospheric corrosion (35). A standard device for measuring humidity, for example, one involving a human hair or by one which detects the presence of moisture condensed on a glass plate or absorbed in a wick, will not necessarily indicate the corrosive responses of a variety of metals with their adherent corrosion products. By way of further complication is the already discussed fact that a given metal will be corroded at different rates in a particular locality depending on the individual conditions of its exposure with regard to orientation and shelter (see, for example, Fig. 7).

For at least the reasons cited, there can be no one quality factor or expression that will indicate the corrosivity of a particular atmosphere towards a single metal let alone any large number of them. Nevertheless, it is possible to make a rough classification of atmospheres based on the corrosion of a particular metal exposed in a particular way for a sufficient length of time (for example a year) to permit seasonal effects to be demonstrated. The most extensive efforts along this line have been made by J. C. Hudson in his work for the British Iron and Steel Institute (29).

Hudson used open-hearth iron and zinc as his test materials. The fact that his investigations were concerned primarily with some quality of the atmosphere rather than of the metals used for test is indicated by the reference to the test pieces as "pollution test" specimens. In his early work, Hudson devoted most attention to his tests with the open-hearth iron specimens, and it is with these tests that we are concerned at this time.

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[7] The aluminum alloy chosen to illustrate the point under discussion is not representative of the most corrosion-resistant aluminum compositions and is not ordinarily employed in corrosive environments without supplementary protection.

In order to eliminate an uncontrolled variable associated with differences in corrodibility of his test pieces, Hudson secured a large number of specimens from the same heat of iron for exposure at the different test sites. When these were used up, additional specimens of about the same composition were secured. There was a difference in copper content of the different specimens of iron, for example, some contained about 0.04 per cent copper and others around 0.07 per cent copper—as will be seen from the data of Fig. 11. This difference in copper content may have had some effect, but probably not sufficient to defeat the purpose of the investigations. However, in any extension of these tests for comparison with Hudson's data, it will be necessary to hold the composition of iron "pollution" test specimens fairly close to the following analysis of the open-hearth iron used most extensively by Hudson:

Carbon, per cent	0.02
Manganese, per cent	0.05
Silicon, per cent	0.008
Phosphorus, per cent	0.013
Sulfur, per cent	0.044
Copper, per cent	0.066
Nickel, per cent	0.054
Chromium, per cent	0.001

Hudson's iron pollution specimens measured 2 by 4 by $\frac{1}{8}$ in. and were exposed vertically at least 5 to 6 ft from the ground and ordinarily faced south. The usual duration of exposure was 1 yr and the loss in weight during this period provided a measure of the "pollution" or corrosivity of the atmosphere at the several test localities.

As a basis of comparison, the most corrosive atmosphere investigated was rated at 100—that of Frodingham near Sheffield, England, where the corresponding weight loss of the standard specimen was 23.0 maximum, 10.37 minimum, 14.81 mean g in 1 yr.

Table VIII provides a listing of several test sites with respect to the corrosivity of their atmospheres as measured in the manner described. Data for some test sites of particular interest to the ASTM have been included for convenient reference. Up to now the most corrosive atmosphere (towards low-copper content open-hearth iron) that has been investigated is that at the test site operated by the author 80 ft from the ocean at Kure Beach, N. C.

TABLE VIII.—RELATIVE CORROSIVITY OF ATMOSPHERES AT DIFFERENT LOCATIONS TOWARDS OPEN HEARTH IRON SPECIMENS 2 BY 4 BY ⅛ IN.

Location	Type of Atmosphere	Average Losses of Weight, g in 1 yr	Relative Corrosivity
Khartoum, Egypt	Dry Inland	0.16	1
Abisco, North Sweden	Unpolluted	0.46	3
Singapore, Malaya	Tropical Marine	1.36	9
Daytona Beach, Fla.	Rural	1.62	11
State College, Pa.	Rural	3.75	25
South Bend, Pa.	Semi-Rural	4.27	29
Miraflores, Canal Zone (Pacific Side)	Tropical Marine	4.5	31
Kure Beach, N. C. (800 ft from Ocean)	Marine	5.78	38
Sandy Hook, N. J.	Marine, Semi-Industrial	7.34	50
Kearny, N. J.	Industrial Marine	7.75	52
Vandergrift, Pa.	Industrial	8.34	56
Pittsburgh, Pa.	Industrial	9.65	65
Frodingham, British Isles	Industrial	14.81	100
Daytona Beach, Fla.	Marine	20.43	138
Kure Beach, N. C. (80 ft from ocean)	Marine	70.49	475

In appraising the reliability of tests of this sort for comparing the atmospheres at different test sites and for measuring changes in corrosivity from time to time at a particular site, it is necessary to pay attention to the following features:

1. The test metal should be responsive

and sensitive to variations in the corrosivity of the atmospheric conditions.

2. The corrosion of the test metal should reflect the effects of climatic conditions throughout the duration of its exposure and should not be influenced too strongly by the conditions that prevail at the start of its exposure period.

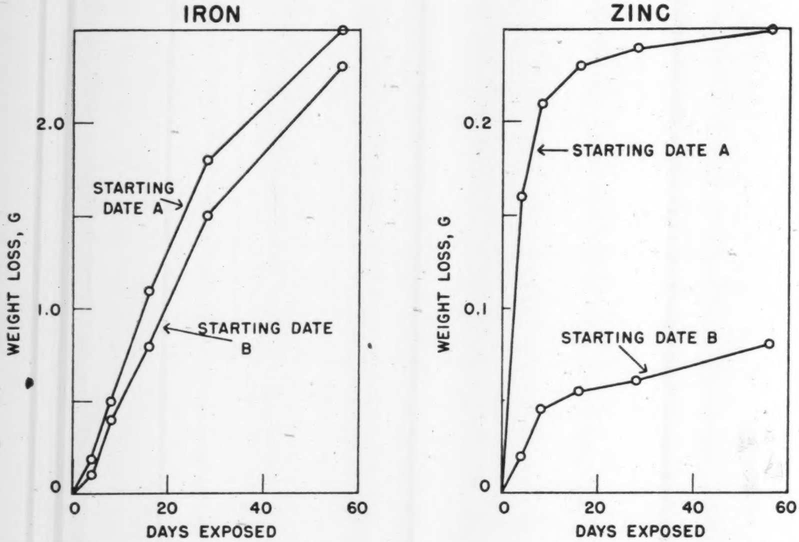


FIG. 20.—Weight Loss *versus* Time for Specimens of Iron and Zinc Started in Test on Different Dates.

Experience with low-copper-content open-hearth iron has indicated that it meets condition 1 quite satisfactorily.

Condition 2 has received a good deal of attention by several investigators. For example, several years ago Schramm and Taylerson (36) reported the results of tests on steel specimens exposed at different seasons of the year and concluded from their data that the extent of corrosion was influenced greatly by whether the weather was wet or dry when the specimens were first exposed. When the test was started in a dry period, the steels were able to develop more protective rusts so that for a given test period their rates of corrosion were substantially lower than those of specimens of the same composition started in test during wet weather. An investigation by Dearden (37) along the same line did not show as great an effect of the climatic conditions at the time of the exposure—probably because during the extended course of his tests the effects of the initial exposure conditions became dissipated.

The most detailed study of the point under discussion was undertaken by O. B. Ellis (38) of the Armco Steel Corp. and described with particular reference to zinc, but with some interesting notes on iron in the paper for which he received the Sam Tour Award in 1950. For zinc, rainfall and relative humidity, especially during the first few hours of exposure, were most significant. For the iron, no particular combination of weather factors could be related systematically to variations in the corrosion that occurred. But, for the purpose under discussion here, Mr. Ellis' data established that zinc was so sensitive to prolonged effects associated with the conditions of its initial exposure as to make it an unsatis-

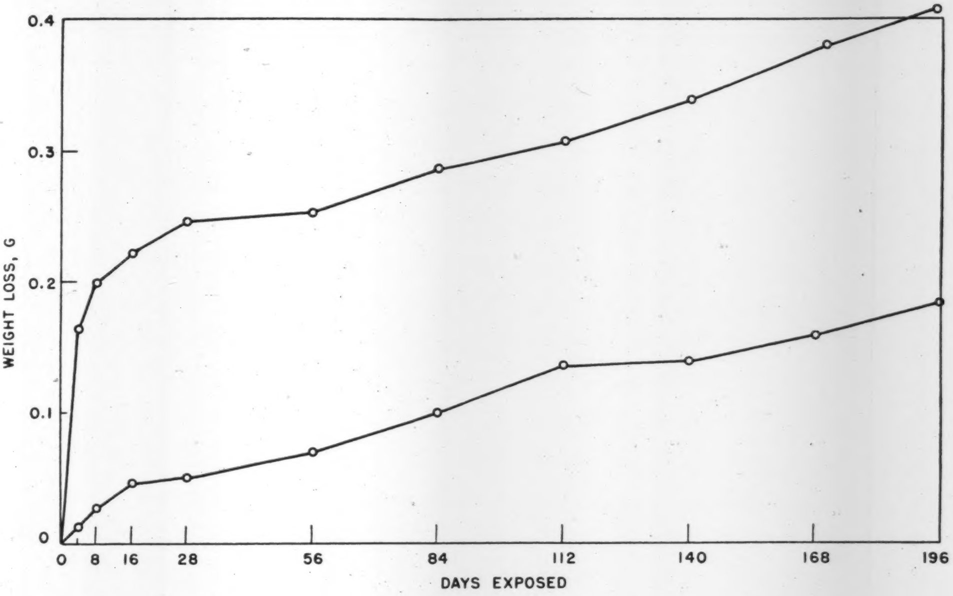


FIG. 21.—Time-Corrosion Curves of Two Samples of Sheet Zinc Showing High and Low Initial Rates of Corrosion.

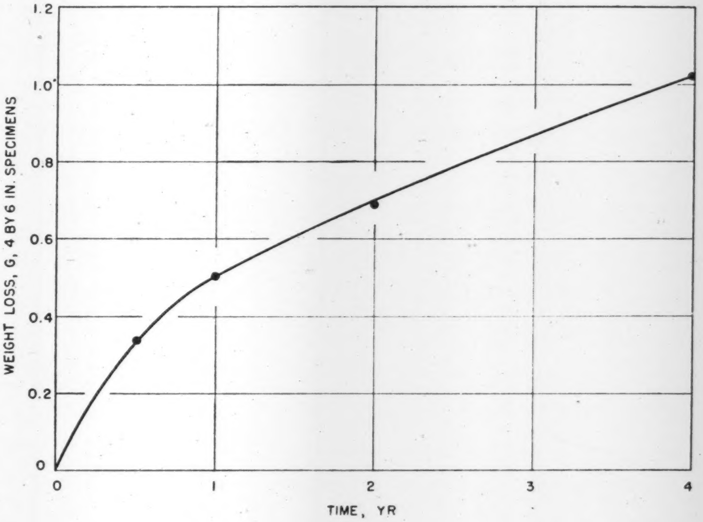


FIG. 22.—Corrosion of Zinc in Marine Atmosphere 800 ft from Ocean at Kure Beach, N. C.

factory material for measuring atmospheric corrosivity. His tests did not prove that iron would be ideal for this purpose—they merely indicated that iron should be preferred over zinc.

Specimens of sheet zinc exposed for consecutive 28 day periods over a period of about 2 yr showed an 8 to 1 variation in the extent of corrosion. Specimens of open-hearth iron exposed at the same times showed a variation in the range of only 1.7 to 1. This difference in sensitivity to peculiar climatic conditions is illustrated by the graphs from Ellis' paper combined in Fig. 20.

Not only is zinc sensitive to climatic conditions as measured by corrosion during the first 28 days of exposure, but the effects of the initial exposure extend over longer periods as illustrated by Fig. 21, which is reproduced from Fig. 9 of Ellis' paper.

It is concluded, therefore, that while iron may not be a perfect material for calibrating the corrosivity of different test sites, it is less sensitive to the peculiar conditions that prevail during the first few days of exposure and is, therefore, to be preferred over zinc for this purpose. This conclusion appears to be contrary to that of British investigators who have exhibited a recent preference for zinc for atmospheric "pollution" tests.

Further light may be thrown on this subject by the results of the tests being made by Subcommittee VII of Committee B-3 in which specimens of iron and zinc are being exposed for different periods at most of the test sites being used by the ASTM for its general atmospheric corrosion testing programs.

Studies of corrosion of zinc in the marine atmosphere at Kure Beach, as illustrated by the data plotted in Fig. 22, have established that in this type of atmosphere, at least, zinc is able to develop corrosion product films that are protective so that corrosion is not a linear function of time. This is contrary to experience in rural, and especially industrial atmospheres where available data from ASTM tests of zinc-coated steel (39) have led to the general conclusion that the life of a zinc coating is directly proportional to its thickness. However, an examination of the data provided by Ellis' tests (Fig. 21) will show that the corrosion of zinc in an industrial atmosphere may also decrease with time during the early stages of exposure. In a strictly marine atmosphere, such as encountered at Kure Beach, a zinc coating twice as thick as another one will last more than twice as long. The practical implication of this is, of course, to give the heavier weight coatings an extra advantage in marine environments.

CORROSION TESTS OF INSECT SCREENS

A practical example where shade or partial shelter has a major influence on the performance of materials is provided by insect screens. In their ordinary use, they are placed in window recesses where they are often shielded from the direct washing action of rain and the drying effects of the sun, yet they are subjected to the corrosive action of condensed moisture—dew—contaminated by sulfur compounds in industrial areas and air borne salt particles near the sea. Under these conditions, many insect screen materials suffer much more severe corrosion than when they are exposed more boldly on ordinary test racks. Surface tension holds films of water and hygroscopic corrosion products bridged across the screen openings, as well as between the wires where they cross, while lint, and other organic matter may act as an absorbent of corrosive liquids. Thus, screen materials encounter more destructive corrosive attack for longer periods than specimens of the same materials exposed boldly in either sheet or wire form.

The net result is that insect screens must withstand atmospheric corrosion in its most extreme forms. Reliable estimates of the probable performance of insect screen materials require that the test specimens be in the form of insect screen and be exposed under the conditions of partial shelter that will be encountered in service. These conditions as to exposure conditions were not wholly satisfied by the tests in which the former ASTM Committee D-14 on Screen Wire Cloth participated several years ago, (40) and, consequently, the results of these tests have restricted application.

A satisfactory method for exposing specimens of insect screen materials was described by W. A. Wesley and H. R. Copson in a paper before this Society in 1946 (41). Their test racks, such as illustrated in Fig. 23, provided for both bold and sheltered exposure. Another feature of their technique was the use of determinations of loss in strength of a representative number of wires taken from the screens after different intervals of exposure as the principal measure of corrosion. This served to measure the weakening effects of localized attack which is so likely to occur at wire intersections and which will not be reflected by weight loss nor be observed readily by visual inspection of fairly resistant materials. Further details concerning this method of test and the effects of partial and complete shelter will be found in the paper mentioned.

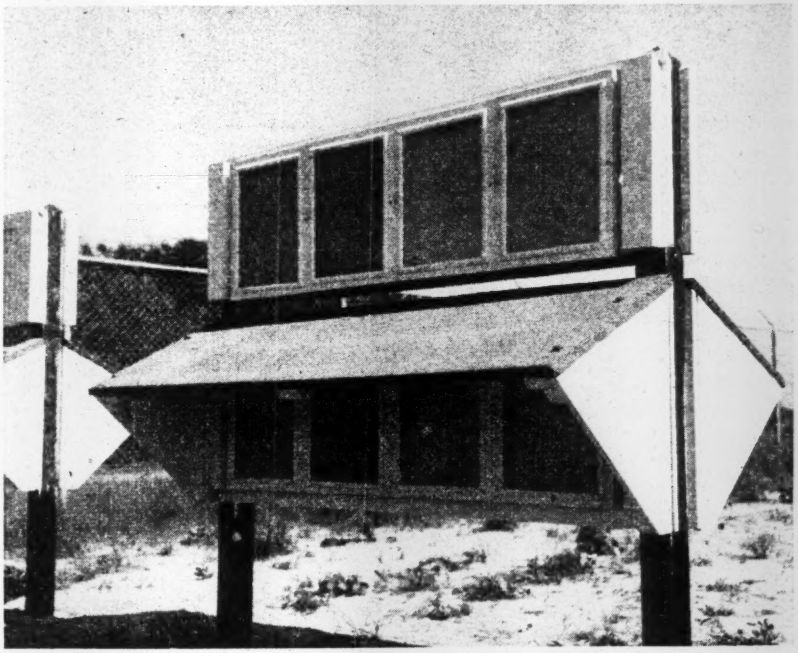


Fig. 23.—Racks Used to Expose Insect Screens Under Condition of Partial Shelter and Bold Exposure.

A similar method of exposure has been used in tests of zinc-coated insect screen in the marine atmosphere at Kure Beach, N. C. With this material, failure occurs rather generally and can be detected readily by visual inspection of the test screens for the appearance of rust and eventual complete destruction of the wires.

The results of one series of these tests illustrate clearly the effects of shelter and orientation on the rate of deterioration of zinc-coated steel insect screens. The screen tested was woven from 0.0115-in. diameter steel wires and was coated with electrodeposited zinc with an average thickness of 0.0003 in.

Specimens were exposed both 80 ft from the ocean facing east and west, and 800 ft from the ocean facing north and south in both boldly exposed and sheltered positions.

The sequence of failure to complete destruction of some wires under the several conditions of exposure was as follows:

1. Facing east towards the ocean, 80 ft from the ocean and sheltered, 187 days (see Fig. 24). Facing east 80 ft from the ocean and unsheltered, 187 days (see Fig. 24).

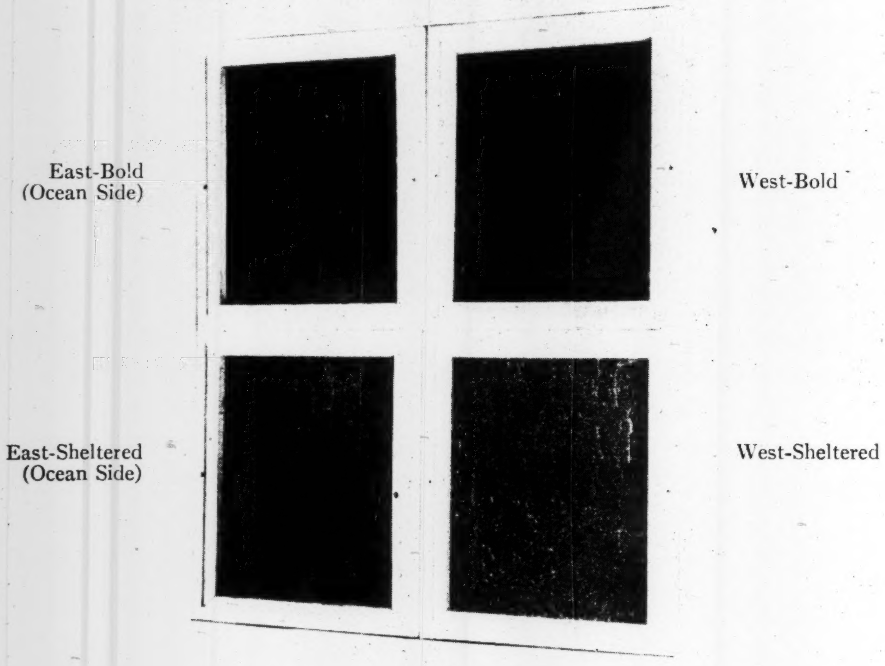
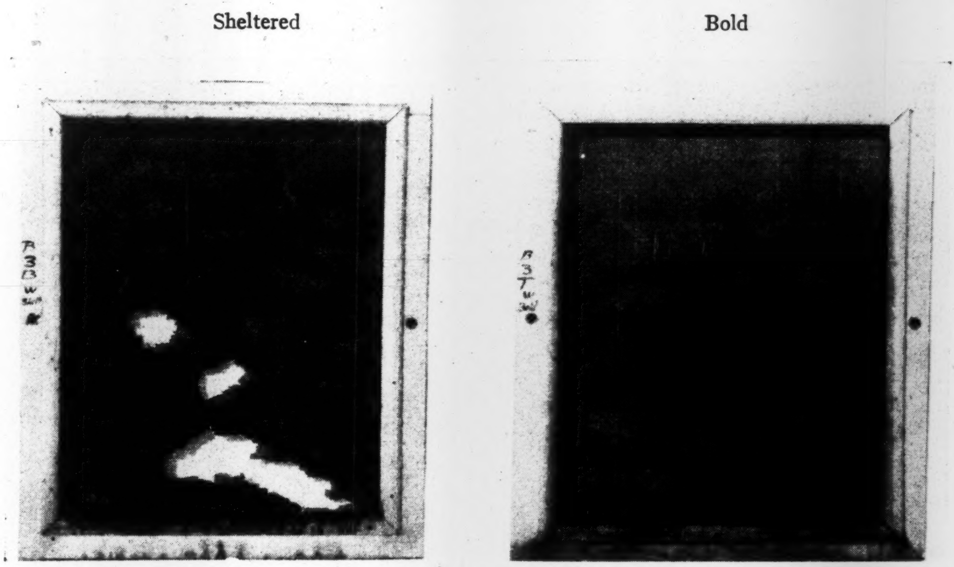
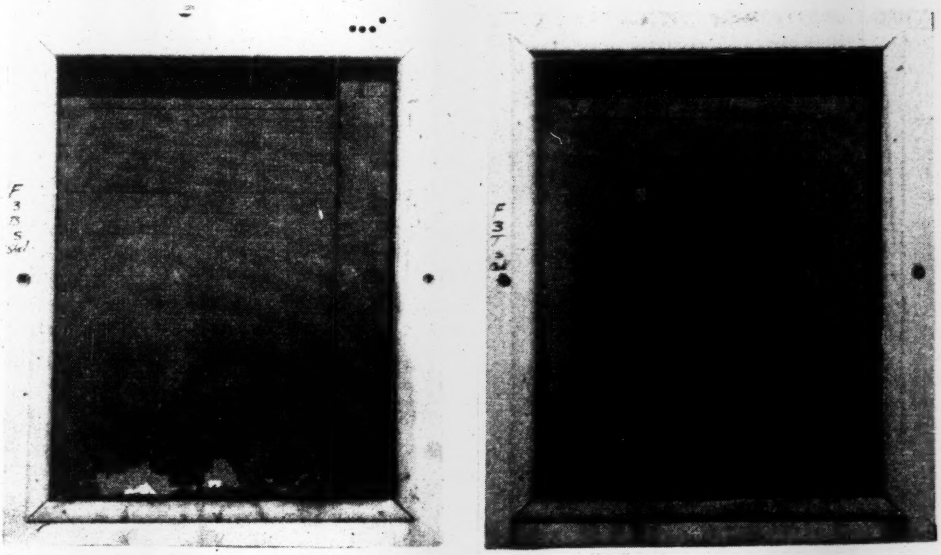


FIG. 24.—Appearance of Zinc-Coated Screens After 187 Days' Exposure in 80 ft Atmospheric Test Lot at Kure Beach, N. C.

2. Facing west away from the ocean, 80 ft from the ocean and sheltered 568 days (see Fig. 25). Facing south 800 ft from the ocean and sheltered, 568 days (see Fig. 25). Facing north 800 ft from the ocean and sheltered, 568 days.
3. Facing west 80 ft from the ocean and unsheltered, 568 days.
4. Facing south 800 ft from the ocean unsheltered, 100 per cent of surface rusted in 568 days, but no wires broken.
5. Facing north 800 ft from the ocean unsheltered, 70 per cent of surface rusted in 568 days, but no wires broken.



80 ft Lot Facing West



800 ft Lot Facing South

Fig. 25.—Appearance of Zinc-Coated Screens After 568 Days Exposure in Atmospheric Test Lots at Kure Beach, N. C.

It will be noted that while the worst conditions of exposure were encountered facing the ocean and only 80 ft from it, the aggravating effect of shelter on the screen exposed this way 800 ft from the ocean was more than enough to make up for its greater distance from the ocean as compared with the boldly exposed specimen 80 ft from the ocean, but facing away from it, which was less severely damaged. All in all, this test illustrates very clearly the dependence of the results on the incidental conditions of exposure with respect to shelter, orientation, and the proximity to a source of corrosive contamination of the atmosphere.

Tests in Waters

There is nothing very complicated about exposing specimens to corrosion by what may be called "natural" waters. The essential features are:

1. To provide for free contact of the specimens with the water with a minimum of shielding by the racks and without setting up regions of peculiar turbulence around the supports.
2. To prevent galvanic disturbances of normal corrosion as a result of contact between specimens of different metals, between specimens and the metal used for the rack, or between specimens and the metal of a structure in the vicinity of the test specimens.
3. To choose a test site where the supply of water will be sufficient or renewed frequently enough to prevent exhaustion of corrosive constituents or contamination with a sufficient concentration of corrosion products to affect the results.

Some suitable designs of test racks and notes on other details of such tests are discussed elsewhere (42) and require no further attention here.

Such tests may be made to discover how different metals may withstand corrosion by the particular water being investigated. In such cases, the data can lead to reasonable and useful conclusions.

But sometimes an object of the test is to study the effects of what might be called minor variations in the composition or structure of materials of essentially the same kind, for example, differend kinds or compositions of steel or iron. In such instances, it is necessary to be very careful not to extend the conclusions to conditions of exposure or of service different from those of the test. The prime reason for this caution is that some external factor of the test environment may exert such a degree of control over the type and extent of corrosion that any possible effects of the internal factors of presumed interest will be smothered or masked to such an extent that they will neither be disclosed nor be disclosable by the results.

TABLE IX.—CORROSION OF DIFFERENT COMPOSITIONS OF IRON AND STEEL WHEN IMMERSED IN SEA WATER AT KURE BEACH, N. C.

Kind of Steel or Iron	Duration of Exposure, yr	Observed Rate of Corrosion, mdd[a]
Carbon steel	6	36
Copper steel	10	33
Hand puddled wrought iron	6	32
2 per cent nickel steel	10	36
1.3 nickel, 0.74 chromium, 0.3 molybdenum steel	8	30
3 per cent chromium steel	10	15

[a] Milligrams per square decimeter per day.

A pertinent example is the simple immersion of specimens in sea water with the object of observing any differences in corrosion resistance between steels or irons of different kind or composition. Results of such tests—with a few notable exceptions—regularly fail to disclose any significant difference in the extent of corrosion of the different steels or irons. Instead, there is an amazing similarity in the extent of corrosion of different kinds of iron or steel varying widely in type and composition as illustrated by the data assembled in Table IX.

The reason for the kind of result illustrated by these data is not that differences in corrodibility do not exist, so much as that when specimens of steel are immersed in sea water the layers of rust and marine growths that accumulate set up a barrier through which oxygen to support the corrosion reaction must diffuse. The rate of this diffusion, which is substantially the same for all the specimens, becomes the major factor in determining how much corrosion can occur—and this amount is then independent of the composition or inherent corrodibility of the steel (43). This conclusion has been reached also by British investigators.

TABLE X.—COMPOSITIONS OF SPECIMENS FROM DIFFERENT SECTIONS OF A RIMMED STEEL INGOT.

Ingot Location	Composition, per cent									
	Nickel	Silicon	Manganese	Copper	Phosphorus	Sulfur	Chromium	Carbon	Molybdenum	Tin
Top core	0.046	0.007	0.38	0.090	0.026	0.069	0.014	0.07	0.021	0.018
Top skin	0.037	nil	0.35	0.074	0.009	0.028	0.018	0.06	0.012	0.011
Middle core	0.038	0.009	0.33	0.074	0.014	0.046	0.014	0.05	0.010	0.014
Middle skin	0.034	nil	0.35	0.063	0.008	0.026	0.031	0.06	0.014	0.012
Butt core	0.038	0.009	0.32	0.068	0.012	0.031	0.017	0.05	0.012	0.014

In the notable case of the chromium steels for which data are included in Table IX, the rust that forms is more impervious than that which forms on most other steels so that the diffusion of oxygen is hindered to a greater extent and a lower rate of corrosion results. It follows, then, that with the maximum rate of corrosion being established by oxygen diffusion rather than corrodibility of the steel it is possible under such conditions to observe rates of corrosion less than the limiting rate, but never higher than it, no matter how fast the steel might corrode if the supply of oxygen or

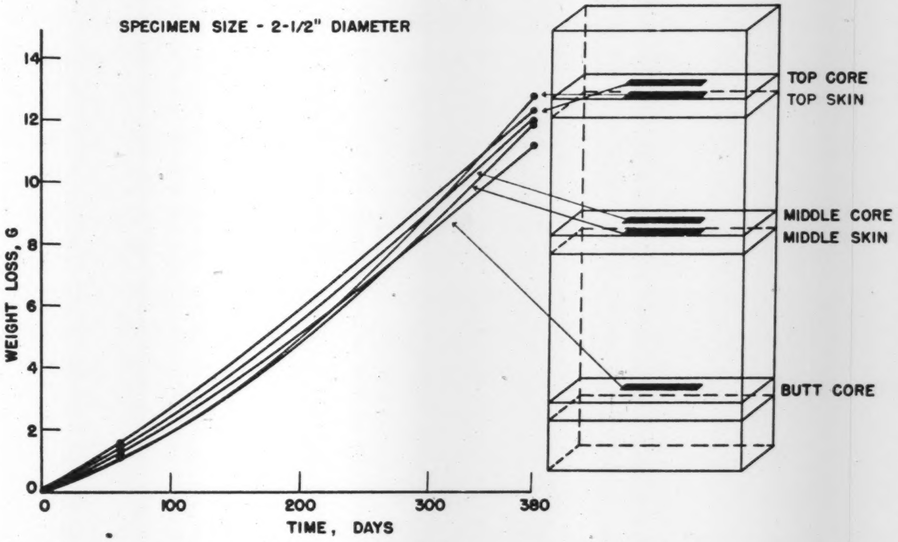


Fig. 26.—Corrosion of Steel From Different Sections of a Rimmed Steel Ingot Exposed to Sea Water Flowing at 1 ft per sec.

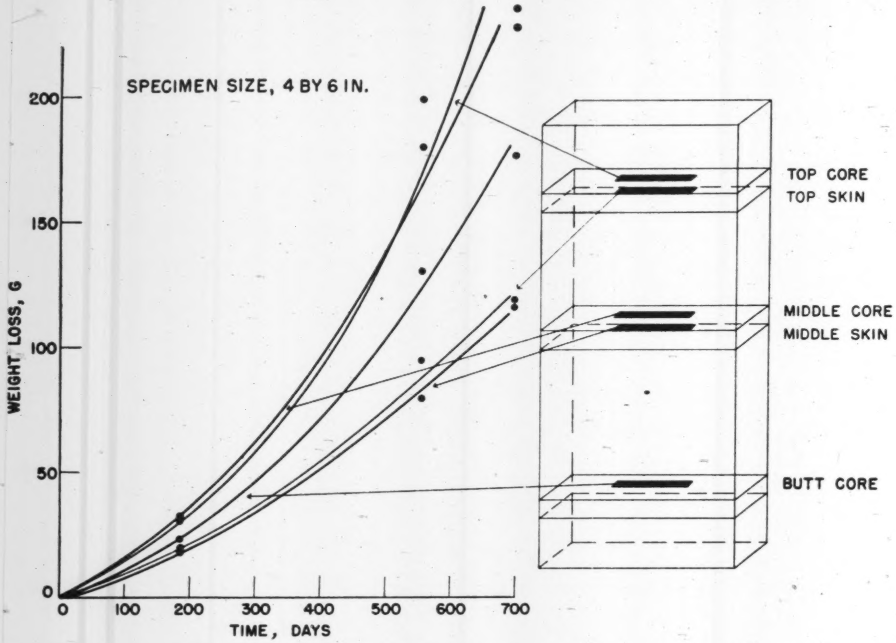


FIG. 27.—Atmospheric Corrosion of Steel From Different Sections of a Rimmed Steel Ingot Exposed 80 ft From Ocean.

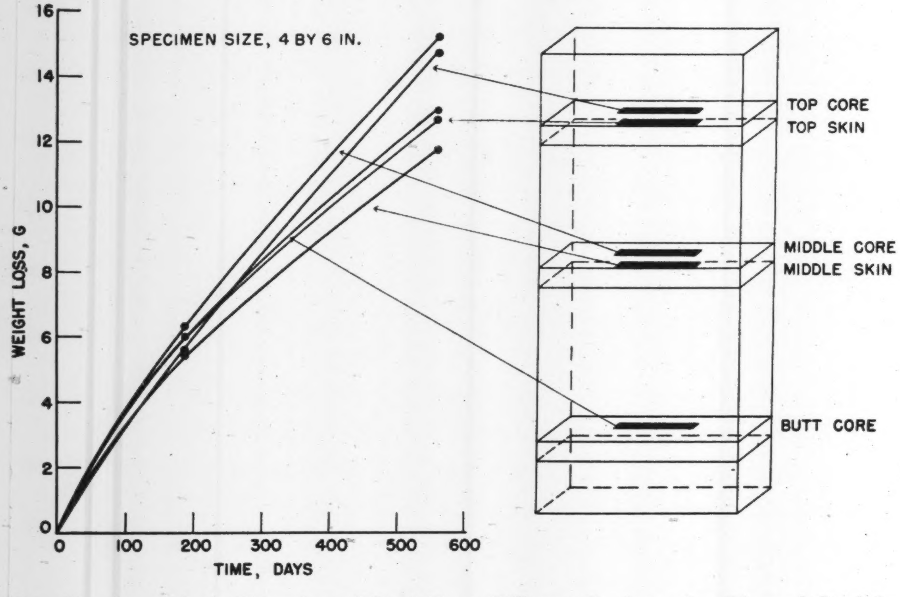


FIG. 28.—Atmospheric Corrosion of Steel From Different Sections of a Rimmed Steel Ingot Exposed 800 ft From Ocean.

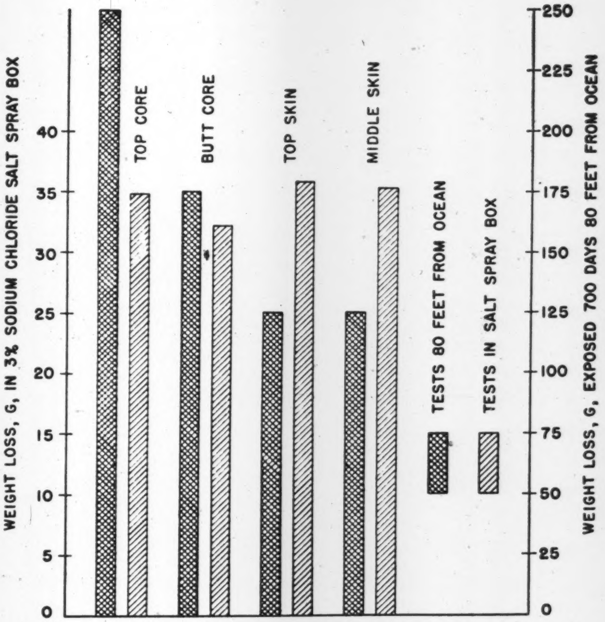


Fig. 29.—Comparison of Results of Salt Spray Test and Natural Exposure of Specimens From Different Sections of a Rimmed Steel Ingot.

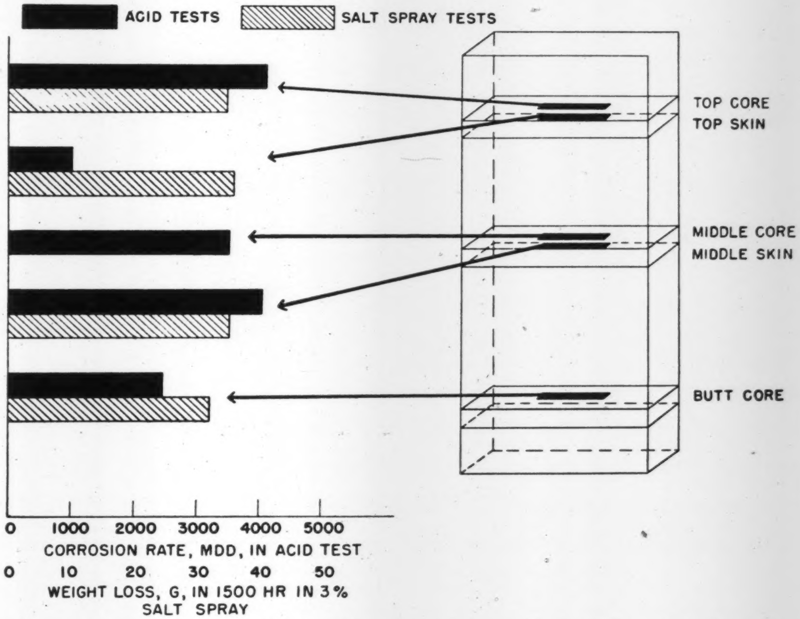


Fig. 30.—Results of Acid and Salt Spray Tests on Specimens From Different Sections of a Rimmed Steel Ingot.

other reactants were not restricted by the external factors.

It is obvious, therefore, that tests of this sort are of little value for appraising any effects of composition or structure on resistance to corrosion in general and particularly on the acceleration of corrosion above normal rates. The achievement of any such objective by the long-time sea water immersion tests carried out by Committee A-5 (**44**) was, therefore, hampered from the start.

Support for the foregoing statements is provided by the case of the chromium steel in Table IX, which showed that corrosion less than that of carbon steel could be disclosed by sea water immersion tests. The failure of such tests to permit observations of differences in corrodibility is illustrated by some interesting tests on specimens representative of different sections of an ingot of rimmed steel.

These specimens were provided by Mr. Clarence Altenburger of the Great Lakes Steel Corp. The relationship between their composition and the sections of the ingot from which they were rolled is shown in Table X.

Groups of specimens were exposed under five conditions:

1. In sea water flowing slowly through a wooden trough for 380 days.
2. On atmospheric test racks 80 ft from the ocean for 554 days.
3. On atmospheric test racks 800 ft from the ocean for 554 days.
4. In a standard 3 per cent salt spray box for 1500 hr.
5. In the standard acid test.

The results are plotted in Figs. 26 to 30. Due to the limiting effects of corrosion product layers and restricted oxygen diffusion in the sea water tests, no differences in corrodibility were observed. Similarly, when exposed 800 ft from the ocean, all of the specimens were able to develop rust films that were about equally protective. But when exposed under the more drastic conditions

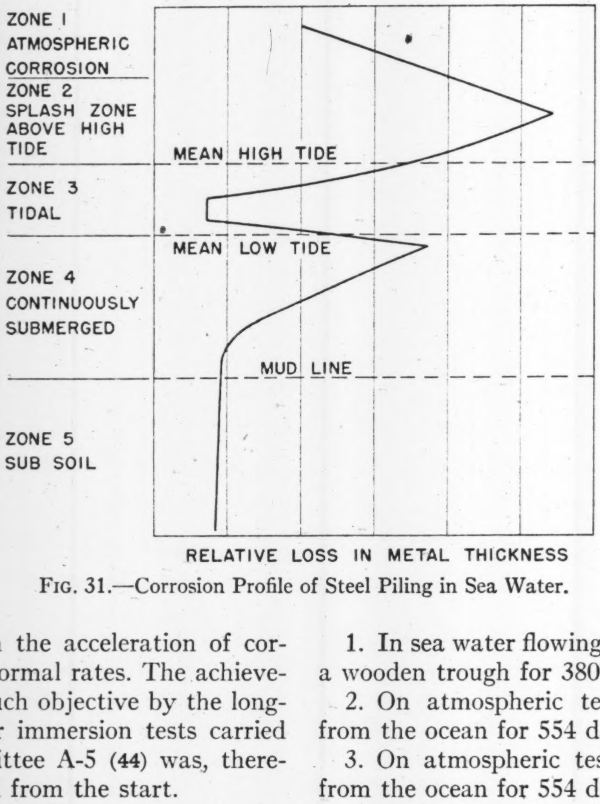


FIG. 31.—Corrosion Profile of Steel Piling in Sea Water.

80 ft from the ocean where the rust films did not become protective enough to exert a controlling effect, the inherent corrodibility or reactivity of the steel from different sections of the ingot had a chance to become apparent and the adverse effect on corrosion of minor constituents, particularly sulfur, was manifested clearly as it was in the acid test. The results of the salt spray test were inconclusive, since in this case they appeared to reflect some controlling barrier effect of the thick layers of rust that formed to about the same extent on all the specimens, rather than either the corrodibilities of the steels or the protective qualities of the kind of rust that formed 80 ft from the ocean.

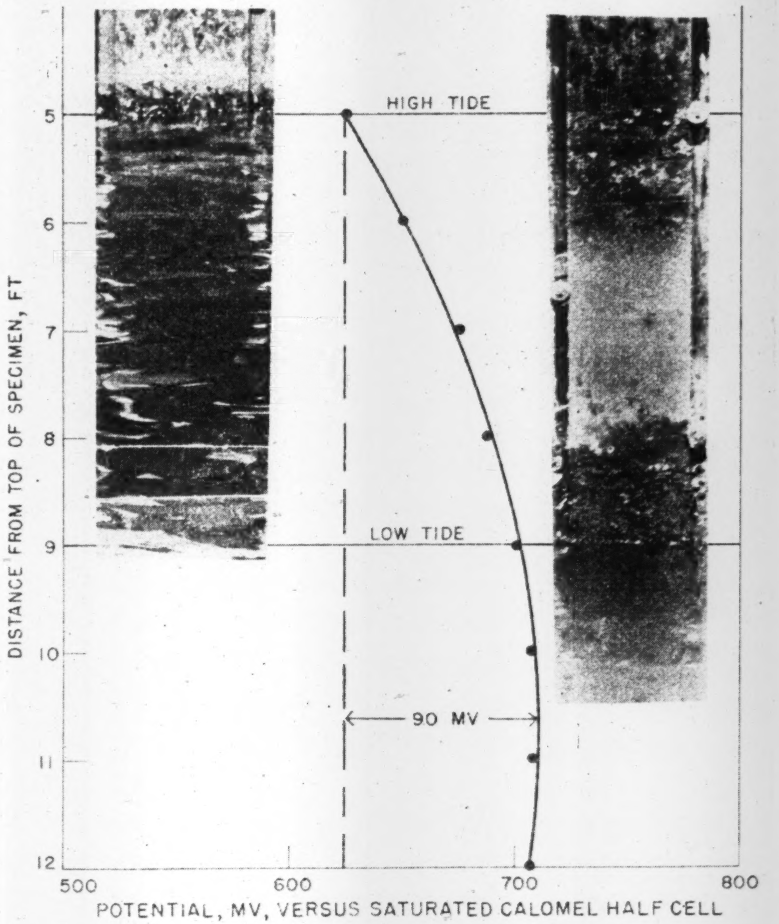


Fig. 32.—Variation in Potential with Depth of Immersion of Steel Plate Extending into the Tidal Zone.

The principle demonstrated is that before drawing conclusions as to effects or lack of them of internal factors, such as composition differences, as indicated by results of corrosion tests, it is necessary to take into account any external factors that can influence the results to such an extent that the effects of any other factors, such as those it is desired to investigate, cannot be noticed.

Before leaving the subject of tests in natural waters, it is necessary to draw attention to some peculiar effects associated with the exposure of steel so that it encounters sea water when continuously immersed under low tide level, alternately wet and dry in the tidal zone, and wet from time to time in the zone of wave action and splash just above high tide level.

Tests of steel in the form of piling which extended through the three zones mentioned yielded a pattern of distribution of corrosion shown in Fig. 31.

The explanation of the low rate of corrosion within the tidal zone was provided by tests by H. A. Humble (45) with groups of coupled and uncoupled steel plates distributed through the three zones of interest.

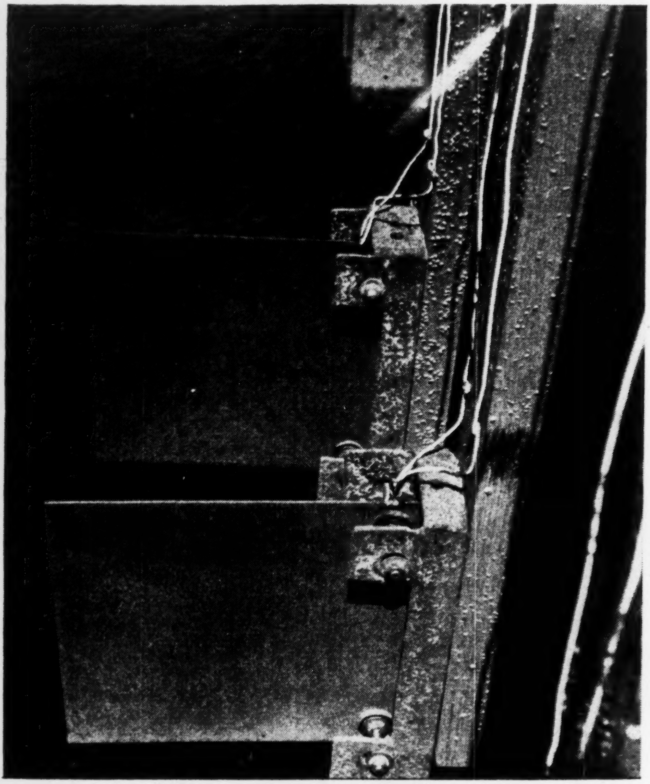


FIG. 33.—Arrangement of Panels in the Tidal Zone Connected Electrically to Submerged Panels.

The key to the situation is that the steel exposed to the highly aerated water in the tidal zone becomes the cathode of a powerful differential aeration or oxygen concentration cell with the anode being the steel below low tide in contact with less well aerated water that seeps through the underwater corrosion products.

It is possible to survey this difference

TABLE XI.—POTENTIAL AND CURRENT MEASUREMENTS BETWEEN STEEL IN THE TIDAL ZONE AND STEEL BELOW LOW TIDE LEVEL (PLATES 12 BY 12 BY ¼ IN.).

	Open Circuit Potential Between Uncoupled Plate (Submerged Plate Anodic), mv	Current Generated Between Coupled Plates, ma
At start	44	21
After 4 months	181	57

in potential by the use of an exploring calomel half cell which can be placed close to the metal surface in the different regions and thus permit measurements of the potentials of the steel at these points. The results of such a potential survey on a steel plate which extended through the tidal zone into the water are shown in Fig. 32.

It is also possible to make similar observations by the technique of Humble using single specimens in the different zones either insulated from each other or connected so as to permit flow of current through a low resistance (1 ohm) shunt introduced to permit measurement of current flow. Such an arrangement is shown in Fig. 33.

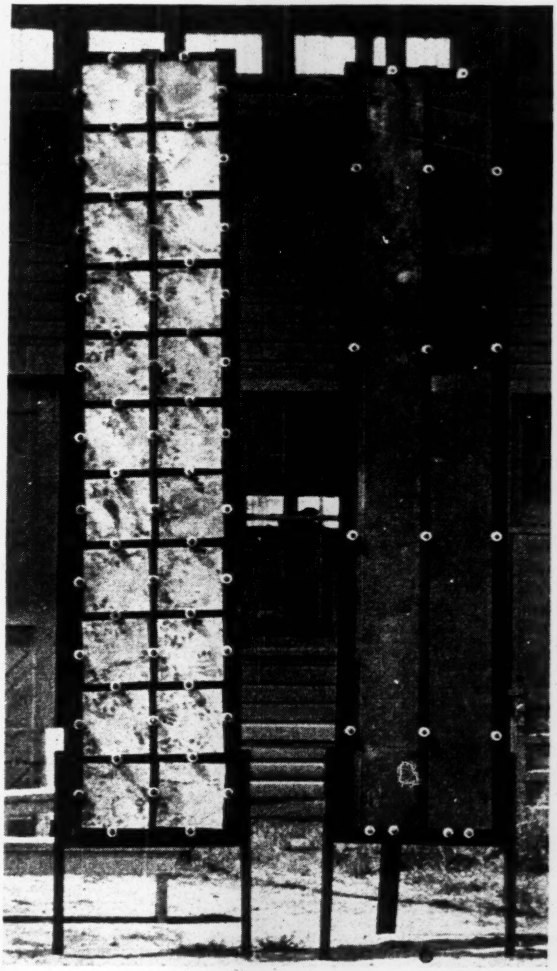


Fig. 34.—Racks and Specimens Before Immersion.

In the case of a continuous plate, or the coupled specimens, the flow of current from the continuously immersed area to the area in the tidal zone causes polarization of the latter so that the usual potential measurements refer to the polarized surfaces and reflect a lower difference in potential than initially exists to energize this naturally powerful local action cell. Some illustrative data from the test arrangement shown in Fig. 33 are assembled in Table XI.

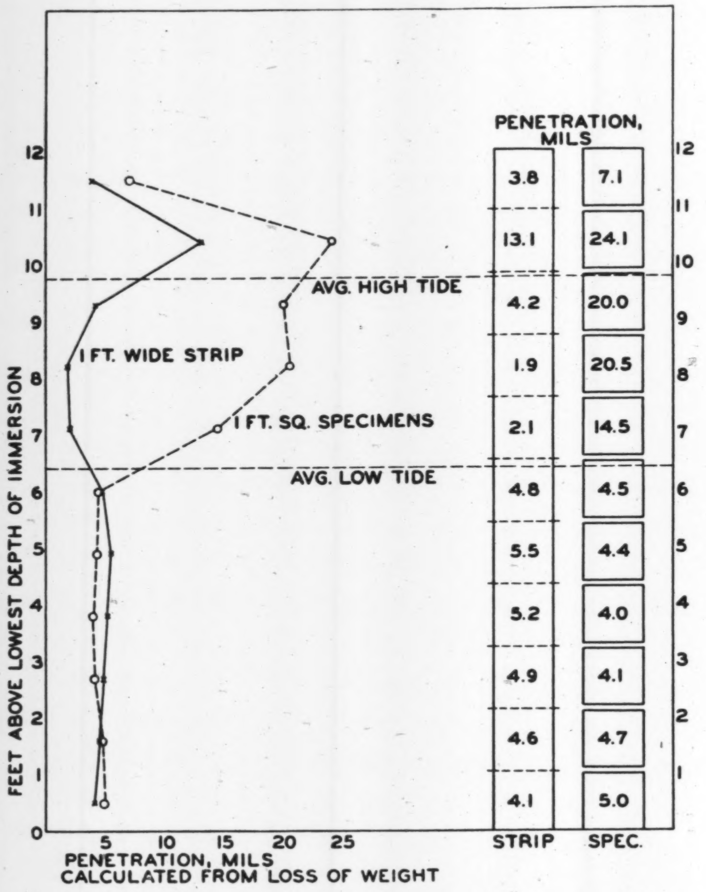


FIG. 35.—Corrosion of Steel Exposed Above, In, and Below Tidal Range (Exposed 151 Days—Kure Beach, N. C.).

Apparently as a result of the oxygen shielding effect of the heavy rust on the submerged steel surfaces, the tendency was for the differential aeration cell to become more powerful and generate more current as the test progressed.

The same effect is illustrated very well by tests of steel in the form of long continuous plates as compared with individual plates of the same steel dispersed through the several zones as illustrated by Fig. 34.

Results of such a test are shown in Fig. 35 from which it will be noted that in the critical region of the tidal zone the rate of corrosion of steel which forms part of a continuous plate is less than one-tenth that of an isolated steel plate exposed at the same location. The difference in appearance of an isolated steel plate and the corresponding portion of a continuous steel plate exposed in the critical tidal zone region is shown in Fig. 36. Note the evidence of the calcareous deposit associated with the self-generated cathodic protection responsible for the low rate of corrosion of the continuous steel plate in the tidal zone. These data have a practical significance with respect to the corrosion and protection of piling and other steel structures partially immersed in sea water. They also are important in disclosing that the practice of studying corrosion in the different zones by exposing isolated specimens in each zone will not provide reliable data applicable to the behavior of structures or components that extend through the several zones. For example, this means that the data for the tidal zone exposures in the long-time sea water tests of the British Institution of Civil Engineers (46) can be applied only to parts which do not extend below low tide and are more generally applicable to exposure in the splash zone where corrosion is usually much more severe than in the tidal zone.

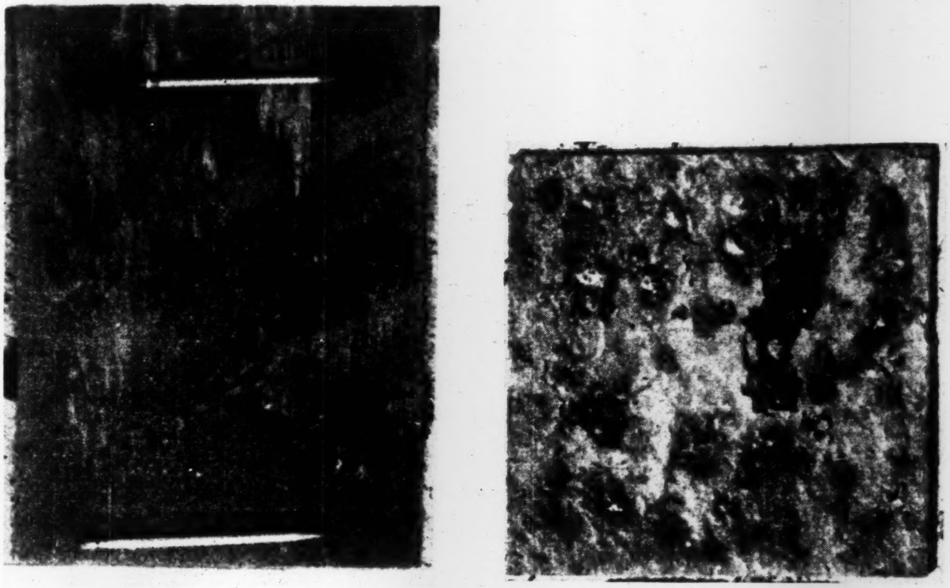

(*a*) Continuous. (*b*) Interrupted.

Fig. 36.—Effect of Depth of Immersion on Corrosion of Low-Carbon Steel After 5 Months' Exposure at Kure Beach, N. C. (Fourth Space From Top, Fig. 34).

It follows, also, that, in some instances, the extent of corrosion of steel in the tidal zone will be influenced by the area of the attached steel below low tide. If this area is small (as when the steel extends barely below low tide) corrosion in the tidal zone will be much more severe than if the submerged area is large (as

TABLE XII.—POTENTIAL AND CURRENT MEASUREMENTS BETWEEN MONEL IN THE TIDAL ZONE AND STEEL BELOW LOW TIDE LEVEL (PLATES 12 BY 12 BY ¼ IN.

	Open Circuit Potential Between Uncoupled Plates, mv	Current Generated Between Coupled Plates, ma
At start	313	41
After 4 months	605	4

when the steel extends well below low tide). It also suggests that even if steel is not needed much below low tide, it might be well to extend the steel in this region for the sole purpose of minimizing corrosion and facilitating protection in the tidal zone.

As illustrated by the data in Table XI, steel in the tidal zone can become considerably more noble than the submerged steel. And, since steel is polarized less readily than some more noble alloys in this zone, it follows that the substitution of a more noble metal for steel in the tidal zone will not necessarily cause any greater corrosion of connected steel underwater.

For example, when a plate of monel is substituted for the steel plate in the tidal zone in the setup shown in Fig. 33 results such as those shown in Table XII are secured.

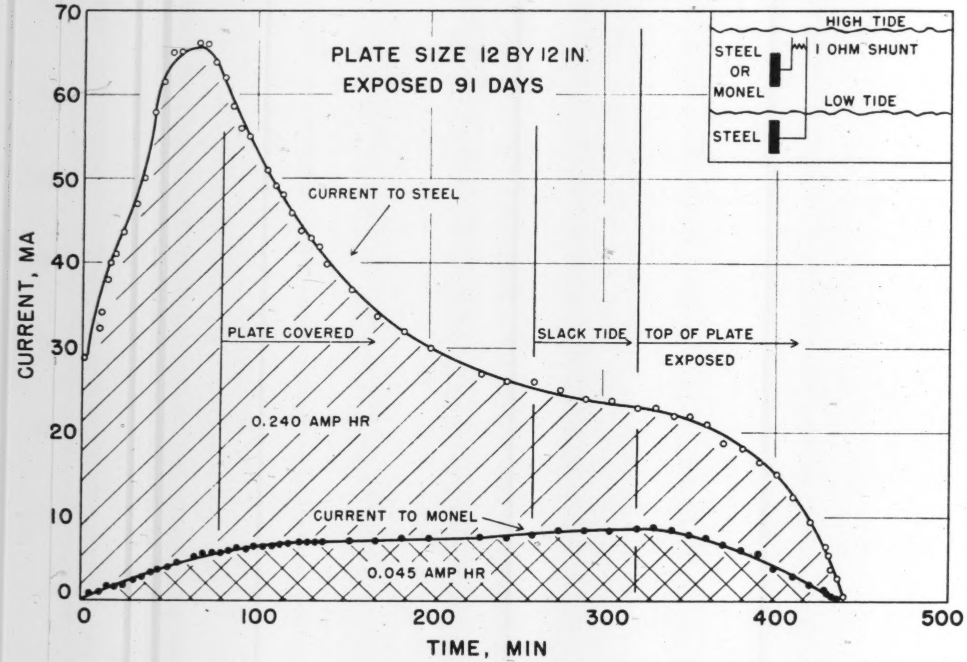


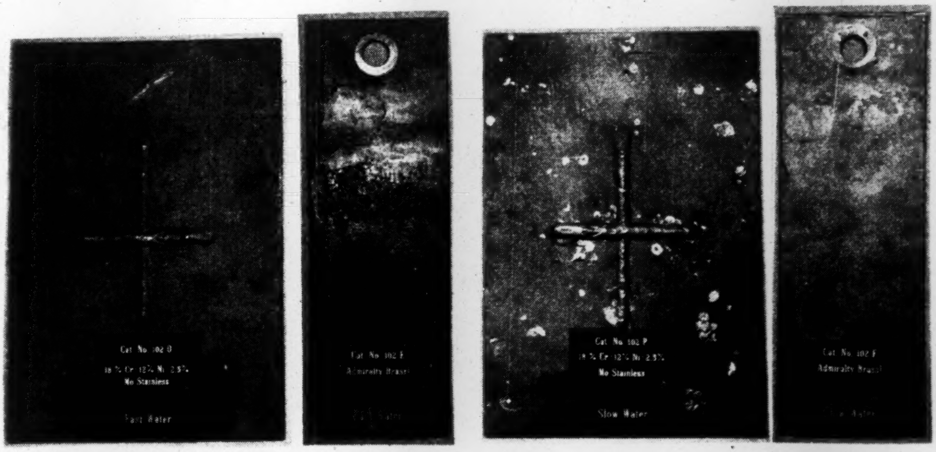
FIG. 37.—Current Flow to Steel and Monel in Tidal Range When Coupled to Submerged Steel.

It will be observed that while the couple with monel developed a greater potential difference than between the two steel plates, the more rapid and more nearly complete polarization of the monel resulted in a great reduction of the galvanic current, so that eventually the steel underwater was corroding less in contact with monel in the tidal zone than when it was coupled to another piece of the same steel in this zone as illustrated by the data in Fig. 37.

This is in line with Humble's discovery

TABLE XIII.—RESULTS OF EXPOSURE OF METAL TO QUIET AND MOVING (4 FT PER SEC) SALT WATER AT FREEPORT, TEX.

Material	Corrosion Rates, mdd		Maximum Pitting, in.	
	Quiet Water	Moving Water	Quiet Water	Moving Water
Admiralty brass	2	24	none	Severely eroded from upstream edge
Type 316 stainless steel	1	0	0.062	none
70–30 cupro nickel	0	0	none	none



18 Cr, 12 Ni, 25 Mo Stainless — Admiralty Brass
(*a*) Specimens in Flume.

18 Cr, 12 Ni,25 Mo Stainless — Admirality Brass
(*b*) Specimens in Basin.

Fig. 38.—Specimens Exposed 1257 Days in Sea Water at Freeport, Tex.

(45) that it was easier to cathodically protect such a monel-steel couple than to protect a steel-steel couple in sea water.

The importance of the incidental conditions of exposure on the results of tests of metals exposed in natural waters, in this case sea water, was illustrated by a series of tests carried out at the plant of The Dow Chemical Co. at Freeport, Tex., on the Gulf of Mexico.

At this plant, salt water from the Gulf is pumped from a collecting basin into a flume through which it flows for use in the several plant processes. At the pumping station a small amount of chlorine, never in excess of 0.5 ppm, is added to the water to suppress the growth of marine organisms in the water-handling system. As a result of this chlorination treatment, plus the effects of the velocity of flow of about 4 ft per sec in the flume, there is little or no growth of barnacles and the like on metal surfaces exposed in this flowing water. On the other hand, there is a heavy growth of organisms on metals exposed to the relatively quiet water in the intake basin.

This situation provided an excellent opportunity for observing the effects of velocity and fouling organisms on corrosion of metals exposed to substantially the same water at the same time. Specimens of several metals were divided by lots into two groups for exposure to corrosion by water under the two conditions described.

The behavior of three alloys—Admiralty brass (70 per cent copper, 29 per cent zinc, 1 per cent tin), Type 316 stainless steel (18 per cent chromium, 12 per cent nickel, 2.5 per cent molybdenum), and iron modified 70–30 cupro nickel (70 per cent copper, 29.5 per cent nickel, 0.5 per cent iron)—will serve to illustrate the great difference in the response of different alloys to the effects of velocity and fouling organisms.

The weight loss and pitting data for specimens of these alloys exposed for 42 months at the two test sites are shown in Table XIII. The equally striking difference in response to the controlling factors is illustrated by the appearances of the brass and stainless steel specimens after test in Fig. 38.

It will be observed that the brass was most vulnerable to attack, especially in the form of erosion of the upstream edge when exposed to the flowing water in the flume. On the other hand, the stainless steel was substantially free from attack under these conditions but suffered severe pitting under the fouling organisms that became attached in the intake basin. The cupro-nickel alloy was much less sensitive to the incidental conditions of exposure and resisted erosion in the flowing water, as well as pitting in the quiet water.

This illustrates very well the fact that conditions of exposure favorable to one class of material may be quite unfavorable to another and *vice versa.* It also shows that it is not possible to classify a material as to the level of its resistance to attack by sea water without specific reference to the incidental conditions of exposure as to velocity, etc., and the type of corrosive attack, for example, erosion or pitting that may occur.

In the case of the stainless steel specimens, the occurrence of attack alongside the welds (weld decay) on the specimens exposed in the quiet water and the absence of similar attack on the duplicate specimens in the moving water is especially noteworthy. Since the disposition of the eight duplicate specimens between the two test sites was by random selection, and since all the specimens were prepared from the same stock and welded by the same welder with the same electrodes at the same time, it may be assumed that the specimens exposed in the flume were sensitized to the same degree and were as susceptible to "weld decay" as those exposed in the basin. Yet, only the latter specimens suffered from "weld decay." This illustrates that susceptibility to intergranular attack—weld decay—may exist without this type of corrosion occurring unless the corrosive nature of the environment is such as to produce this effect. A variation in corrosive nature due simply to a change in velocity of the corrosive medium may be sufficient to represent the difference between considerable intergranular attack and none at all. This has an obvious bearing on the general significance of arbitrary tests, such as in boiling nitric acid, in determining susceptibility to intergranular attack as affected by composition and heat treatment.

GALVANIC CORROSION

The frequent practical necessity of using metals and alloys in electrical contact with each other in equipment and structures subjected to the action of corrosive electrolytes is the basis for considerable interest in the galvanic acceleration of corrosion that may occur.

The mechanisms involved in galvanic corrosion are extremely complicated and appear to become more rather than less so as the subject is investigated more intensively. But it is a common characteristic of galvanic corrosion that the attack of one of the metals in the couple is increased while that of the other is reduced as a result of the galvanic action. Whatever the extent of this galvanic action may be, one of the things that it is desirable to know in advance of actual experience is which of the metals in a proposed couple is likely to suffer and which to benefit from the galvanic effect.

An answer to this simple question will be provided by an arrangement of the common metals and alloys in what can be called a galvanic series. Such a series is based primarily on the measurement of the relative electrical potentials of the several metals when exposed to the corrosive environment of interest. Such differences in potential may be determined directly, or the potentials of the individual metals may be measured relative to some reference such as a calomel half cell. The potentials are influenced by the same internal and external factors that control other manifestations of corrosion and are, therefore, not invariable characteristics of a given metal in a given environment. They also change as a result of polarization[8] effects associated with the flow of the current that produces the galvanic action. It is customary, therefore, in making the potential measurements to take great

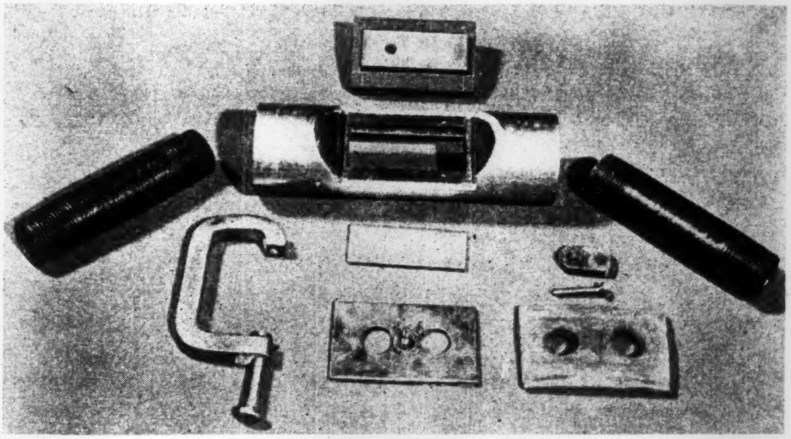


Fig. 39.—Apparatus for Potential Measurements in High-Velocity Sea Water (Disassembled).

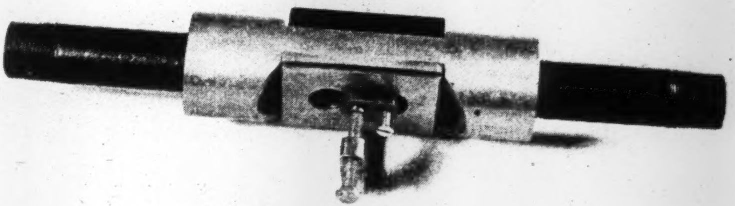


Fig. 40.—Apparatus for Potential Measurements in High-Velocity Sea Water.

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[8] For the purpose of this discussion, polarization is defined as the change in the measured potential of a metal surface in an electrolyte as the result of the flow of current to or from it. In the case of anodic polarization (current flow from the metal to the solution) the potential becomes more noble, and with cathodic polarization (current flow to the metal), becomes less noble. In galvanic couples, the effect of polarization is to bring the potentials of the coupled metals closer together.

precautions to restrict the flow of current during the measurements and thus avoid the polarization that influence the measurements. It is obvious, that such open circuit potentials have only a limited relationship to the polarized potentials that determine the extent of current flow when galvanic corrosion is actually occurring. It is equally obvious that predictions of the probable magnitude of galvanic corrosion solely on the basis of open circuit potential measurements are likely to be quite unreliable. About all that the potential measurements indicate is the probable direction of the flow of galvanic currents and consequently which of the metals in the couple will experience acceleration of its corrosion and which retardation of its corrosion. It must also be remembered that potential relationships established for one corrosive environment will not necessarily extend to another or even to the same corrosive medium under different incidental conditions of exposure with respect to such factors as aeration, temperature and velocity.

While drawing attention to these limitations of the data provided, the author of the present paper, with his colleague Mr. G. L. Cox, presented a paper (47) before this Society at its 1940 meeting which included an arrangement of several metals and alloys in a galvanic series based on observations of their relative potentials in slowly moving sea water.

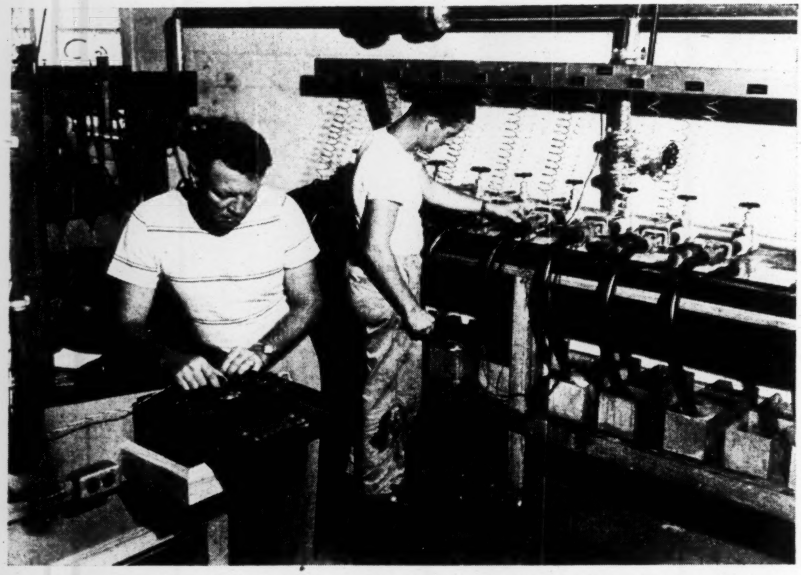


FIG. 41.—Cells for Polarization Studies in Flowing Sea Water.

Since the metals included in this series maintain roughly the same relationship in many other environments, this series has been used as the basis for estimating the probable direction of galvanic effects in other environments for which specific data have been lacking.

More recently the author and his associates at the International Nickel Marine Corrosion Testing Laboratories at Kure Beach and Harbor Island, N. C., have been using a new design of apparatus which permits the measurement of galvanic potentials and currents over a wide range of velocity of flow of sea water over the surfaces of the test specimens.

The essential details of this apparatus are shown in Figs. 39 and 40 and the arrangement of a battery of these test cells with the accessory electrical control panels for polarization studies in Fig. 41.

The most important advantages of this apparatus are:

TABLE XIV.—ARRANGEMENT OF METALS AND ALLOYS IN A GALVANIC SERIES BASED ON POTENTIAL MEASUREMENTS IN SEA WATER. VELOCITY OF FLOW, 13 FT PER SEC; SEA WATER TEMPERATURE, 25 C (77 F).

Metal	Steady Potential Negative to Saturated Calomel Half Cell, v
Zinc	1.03
Aluminum (Alclad 3S)	0.94
Aluminum 3S-H	0.79
Aluminum 61 S-T	0.76
Aluminum 52 S-H	0.74
Cast iron	0.61
Carbon steel	0.61
Stainless steel type 430 (17 per cent chromium)[a]	0.57
Ni-Resist cast iron (20 per cent nickel)	0.54
Stainless steel type 304 (18 per cent chromium 8 per cent nickel)[a]	0.53
Stainless steel type 410 (13 per cent chromium)[a]	0.52
Ni-Resist cast iron (30 per cent nickel)	0.49
Ni-Resist cast iron (20 per cent nickel + copper)	0.46
Naval rolled brass	0.40
Yellow brass	0.36
Copper	0.36
Red brass	0.33
Composition G bronze	0.31
Admiralty brass	0.29
90-10 cupro nickel (0.8 per cent iron)	0.28
70-30 cupro nickel (0.06 per cent iron)	0.27
70-30 cupro nickel (0.47 per cent iron)	0.25
Stainless steel type 430 (17 per cent chromium)[a]	0.22
Nickel	0.20
Stainless steel type 316 (18 per cent chromium, 12 per cent nickel, 3 per cent molybdenum)[a]	0.18
Inconel	0.17
Stainless steel type 410 (13 per cent chromium)[a]	0.15
Titanium (commercial)	0.15
Silver	0.13
Titanium (high purity from iodide)	0.10
Stainless steel type 304 (18 per cent chromium, 8 per cent nickel)[a]	0.08
Hastelloy C	0.08
Monel	0.08
Stainless steel type 316 (18 per cent chromium, 12 per cent nickel, 3 per cent molybdenum)[a]	0.05

[a] The stainless steels as a class exhibited erratic potentials depending on the incidence of pitting and corrosion in the crevices formed around the specimen supports. The values listed represent the extremes observed and due to their erratic nature should not be considered as establishing an invariable potential relationship amongst the alloys covered.

1. The test specimens are stationary and are in contact with the sea water on only one surface—thus facilitating making and maintaining electrical connections on the surface outside the cell.

2. The tip of the reference calomel half cell may conveniently be brought close to the surface being studied when measurements are to be made and withdrawn so as not to disturb the flow of water or interfere with the flow of current at other times. (This is accomplished simply by inserting the tip of a half cell through a hole in the specimen or electrode that is opposite the specimen being studied.)

TABLE XV.—RELATIONSHIP BETWEEN OPEN CIRCUIT POTENTIALS AND CORROSION RATES OF METALS IN SEA WATER FLOWING AT 13 FT PER SEC AT 25 C (77 F).

Metal	Steady Potential Relative to Saturated Calomel Half Cell, v	Corrosion Rates Calculated from Weight Losses, mdd
Zinc	1.03	1490
Aluminum 3S-H	0.79	45
Carbon steel	0.61	325
Naval rolled brass	0.40	24
Copper	0.36	106
Nickel	0.20	55
Type 410 stainless steel (13 per cent chromium)	0.15	114
Hastalloy C	0.08	2

3. The desired rates of flow of sea water can be measured and regulated readily.

4. Uncontaminated sea water is available under a constant hydraulic head in the large quantities needed. The battery of 22 test units requires a total of 300,000 gal per day.

This apparatus has been used to provide the data for a galvanic series based on open circuit potential measurements in highly turbulent sea water flowing at a calculated velocity of 13 ft per sec. This will supplement the data reported previously (47) for sea water flowing at a velocity of only about $\frac{1}{4}$ ft per sec.

The newly determined potential values as shown in Table XIV represent what were considered to be the "steady state" potentials as measured usually at the end of a 14-day period of exposure.

The relationships between these potential values and resistance to corrosion as reflected by weight loss for some typical materials are shown by Table XV.

It is obvious, from these data, that open circuit potential measurements are quite unreliable for predicting resistance to corrosion even on a relative basis. A reason for this is that the potentials as they are usually measured are simply the average polarized potentials of the anodes and cathodes in the local action cells that cause the corrosion that is observed. Depending on the cathodic and anodic polarization characteristics of the metal, a measured potential may be associated with either a very high or very low anodic current density, as illustrated by the electrode potentials in a typical local action cell in Fig. 42.

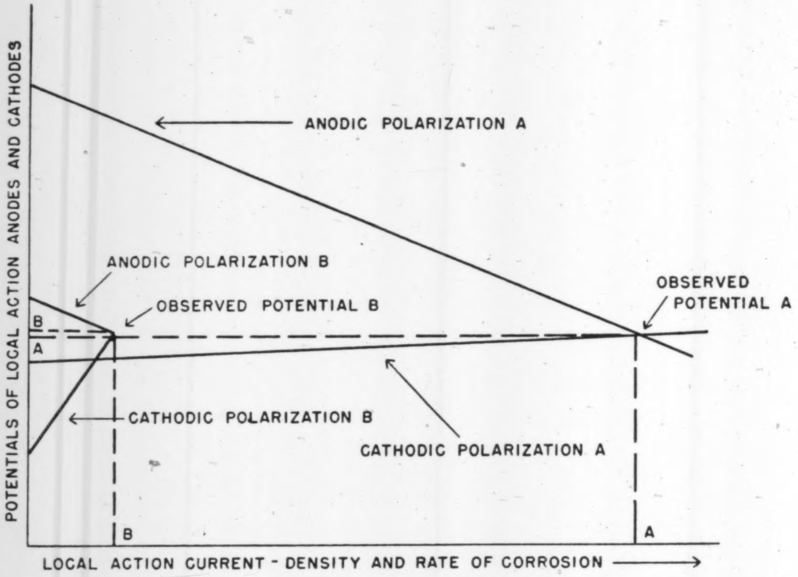

POTENTIALS OF LOCAL ACTION ANODES AND CATHODES
ANODIC POLARIZATION A
ANODIC POLARIZATION B
OBSERVED POTENTIAL B
OBSERVED POTENTIAL A
B
A
CATHODIC POLARIZATION B
CATHODIC POLARIZATION A
B
A
LOCAL ACTION CURRENT - DENSITY AND RATE OF CORROSION

FIG. 42.—Relationship Between Observed Potentials, Polarization, and Corrosion Rates.

In each case, the measured potential will be that established by the intersection of the anodic and cathodic polarization curves. With a metal like A, where cathodic polarization is negligible, the local action current density associated with the intersection of the anodic and cathodic polarization curves is high and the rate of corrosion is correspondingly high. However, with metal B, cathodic polarization is considerable so that the intersection of the anodic and cathodic polarization curves occurs at a low current density and with a corresponding low rate of corrosion. Nevertheless the open circuit potentials of the local anodes and cathodes taken in conjunction with the polarization characteristics described can be such that the average open circuit potentials as usually measured may be almost alike, yet associated with gross differences in the magnitude of the local action currents and corresponding corrosion rates.

This discussion of polarization in local action cells suggests their similarity to galvanic couples of dissimilar metals where the anodic reaction occurs predominantly on one metal and the cathodic reaction on the other.

It is evident, therefore, that the polarization characteristics of metals are of major importance not only in connection with normal corrosion but especially in galvanic corrosion where, in the nature of things, there are well-defined anodes and cathodes. In many, if not most, practical cases of galvanic corrosion the most important controlling factor is the extent of polarization of either the anode or the cathode or both. Furthermore, in many practical cases of galvanic corrosion it is the polarization of the cathode (the more noble metal in the couple) that is the major controlling factor. Consequently, the study of cathodic reactions and how they occur so as to influence cathodic polarization of different metal surfaces is of special importance in investigations of galvanic corrosion.

It was very fitting, therefore, that at the time the author and G. L. Cox presented their paper on galvanic potentials in sea water (47) there was an important companion paper by their associate W. A. Wesley (48) who focused attention on the importance of the polarization effects that have been mentioned here. In the light of our experiences since Wesley's paper was published, as illustrated by some typical data to be described here, our knowledge of how metals may behave in galvanic couples would be much greater and more precise than it is now if more attention had been given during the past ten years to studies of polarization phenomena as suggested by Wesley's work at that time. It follows also that this is an avenue of corrosion research that promises a maximum yield from an effort to improve our understanding of corrosion phenomena and the fundamental knowledge of how and why metals corrode.

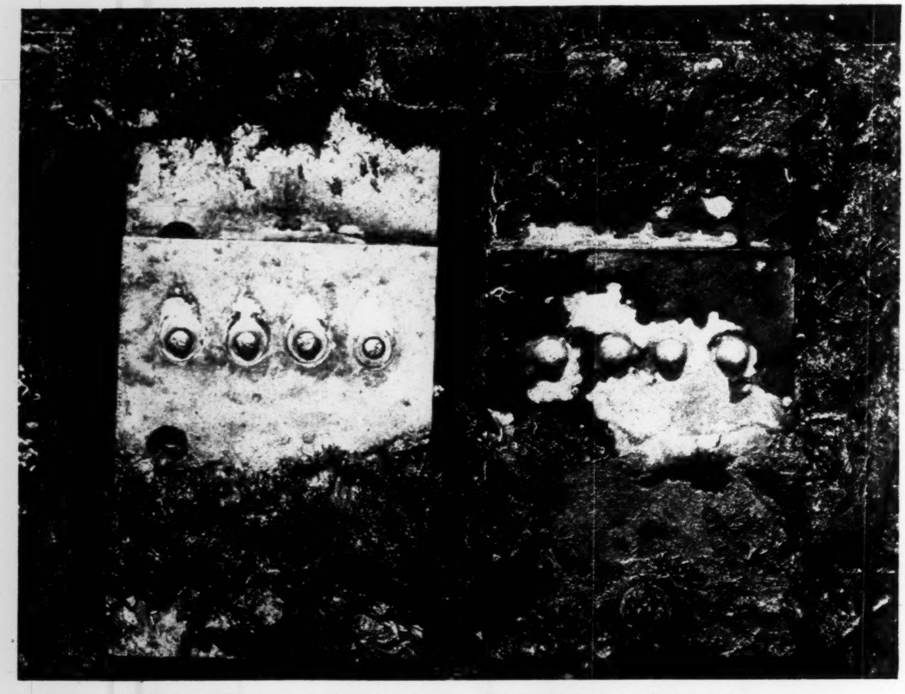
It will suffice, here, to provide some simple illustrations of what we are talking about and to demonstrate how little we actually know about what may happen when we have determined only the open circuit potentials of the metals in which we are interested and have arranged them in a galvanic series such as is provided by Table XIV.

The first example involves no more than the "area effect" which was illustrated quantitatively by the experiments of Whitman and Russell (49) and later rationalized on the basis of polarization effects by Wesley (48).

As would be expected, the extent of polarization increases with current density, so that with a given amount of current flowing from an anode—such as steel joined to copper—the current density on a large piece of copper will be less than on a small piece of copper. The extent of polarization of the copper in the former case will be less than in the latter. For a quantitative illustration of this effect see paper by W. A. Wesley (48). This means that in operating galvanic cells the polarized working potential of a cell made up of a small piece of steel joined to a large piece of copper will be much higher than the polarized working potential of a cell made up of a large piece of steel joined to a small piece of copper—even though the open circuit potential will be the same in each case.

A corollary effect in the examples cited is that with a small piece of steel the anodic current density will be high, and with a large piece of steel it will be low for the same amount of total current. And, since the rate of corrosion is proportional to current density, the greatest amount of destruction from galvanic corrosion will usually be associated with circumstances where the galvanic couple is made up of a small anode and a large cathode, while the reverse situation may produce a negligible galvanic effect.

A simple practical example of the area effect is provided by rivet and plate assemblies. Steel rivets in a copper plate represent a dangerous combination of a small anode (steel) and a large cathode (copper), while the reverse arrangement of copper rivets in a steel plate represents a relatively safe combination of a large anode and a small cathode.



(*a*) Copper Plates, Steel Rivets. (*b*) Steel Plates, Copper Rivets.
FIG. 43.—Area Effect Shown by Rivet and Plate Assemblies.

The validity of this principle is illustrated by the specimens shown in Fig. 43.

The undesirable area relationship favored the rapid corrosion of the steel rivets used to joint two copper plates at the left in Fig. 43, while with the desirable area relationship in the steel plates joined by copper rivets at the right in Fig. 43 there was no significant acceleration of the corrosion of the steel near the copper rivets and of course no corrosion of the copper rivets themselves.

If all metals were subject to cathodic polarization to the same extent under the same conditions of current flow, the study and prediction of galvanic effects would be much simpler than it is. However, different metals act quite differently with respect to their influence on the reactions that occur on their surfaces and which are responsible for the polarization that occurs.

The principal reactions (50) that occur at cathodic surfaces are:

1. $2H^+ + 2e^- = H_2$ (Evolution of hydrogen).

2 $O_2 + 4H^+ + 4e^- = 2H_2O$ (Reduction of oxygen to water).

3. $O_2 + 2H^+ + 2e^- = H_2O_2$ (Reduction of oxygen to hydrogen peroxide).

It has long been known that there are gross variations amongst metals with respect to their influence on reaction 1—the evolution of hydrogen. On some surfaces, the reaction occurs readily and on others reluctantly. The differences can be measured in terms of the hydrogen overvoltage values of the different metal surfaces. These values are also dependent on the current density involved, and tables of overvoltage values for different current densities may be found in reference books.

It has also been found that different metals have different effects on reactions 2 and 3 involved in oxygen ionization or oxygen reduction (sometimes referred to as hydrogen oxidation). Evans, Bannister, and Britton (51) showed that these reactions occurred more or less readily on different oxide films with consequent effects on cathodic polarization.

Tomashov (52, 53) has endeavored, by polarization studies, to assign oxygen reduction overvoltage values at different current densities to different metal surfaces. More recently, similar studies have been made by Delahay using an improved polarographic method (54).

While the amount of work that has been done by these investigators has not permitted the tabulation of precise values for oxygen reduction overvoltages, it has been sufficient to show that gross variations amongst metals do exist and have their influences on the cathodic polarization reactions and the shapes of cathodic polarization curves.

Direct support of these facts is provided by the results of some recent studies of the cathodic polarization characteristics of some common metals and alloys in flowing sea water undertaken by the author and his associates. The apparatus used was that previously described and illustrated in Figs. 39 and 41.

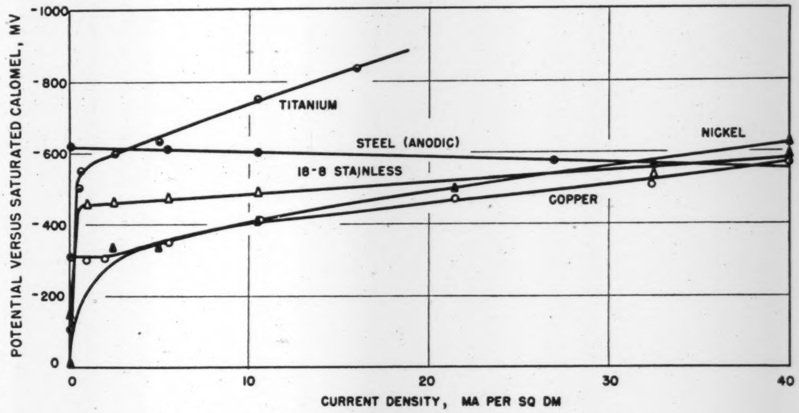


Fig. 44.—Polarization of Metals in Flowing Sea Water.

For the polarization studies, current at controlled densities was applied to specimens of the metals under investigation from platinum anodes which formed one side of the sea water channels while the specimens under study made up the other. A single specimen was used for each current density investigated and the current was held constant throughout the course of each test, which usually ran for about a week.

Typical cathodic polarization curves determined in this manner for titanium, copper, 18-8 stainless steel, and nickel, combined with an anodic polarization curve for steel, are shown in Fig. 44.

Neglecting resistance effects, the current densities corresponding to the points of intersection of the anodic polarization curve for steel and the cathodic polarization curves for the other metals should provide a fair measure of the relative extents by which the corrosion of steel would be accelerated when placed in galvanic contact with each of the other metals.

It is evident that each of the metals has its own cathodic polarization characteristics in flowing sea water, determined presumably by its effects on the cathodic reactions of hydrogen evolution or oxygen reduction as previously discussed. We might say, for example, that these reactions occur readily on a copper surface and only sluggishly on a titanium or stainless steel surface.

TABLE XVI.—GALVANIC CORROSION OF STEEL IN CONTACT WITH DIFFERENT METALS IN SEA WATER FLOWING AT 7.8 FT PER SEC AT 45 F FOR 16 DAYS.

Couple	Open Circuit Potentials in Volts *versus* Calomel Half Cell						Internal Resistance of Cells, Ohms	Current Observed During Test, ma			Rate of Corrosion of Steel In Couple, mdd	Galvanic Effect, mdd	Rate of Corrosion of Other Metal in Couple, mdd
	At Start			At Finish[a]									
	Steel	Other Metal	Difference	Steel	Other Metal	Difference		Max	Min	Avg			
Steel to steel	0.509	0.502	0.007	0.610	0.596	0.014	1.4	1.28	0.22	0.44	183	28	156
Steel to copper	0.515	0.286	0.229	0.597	0.241	0.356	2.3	4.23	2.10	3.2	525	355	10
Steel to stainless steel (18-8)	0.502	0.177	0.325	0.577	0.071	0.506	2.9	0.40	0.22	0.31	195	25	0.7
Steel to titanium	0.502	+0.010	0.512	0.596	0.031	0.565	2.6	0.47	0.19	0.39	224	54	1.0
Steel to nickel	0.533	0.151	0.382	0.595	0.097	0.498	3.0	8.43	1.10	3.6	607	437	2.2

[a] Measurements made when metals had become depolarized in about 3 hr after circuits were broken.

In order to see whether the indications of the polarization curves would be realized in practice, some galvanic couple corrosion tests were carried out in the apparatus that was used for the polarization studies.

In these tests, specimens of steel were placed in the test units in electrical contact with specimens of each of the other metals. A couple of steel to steel was also included. The electrical connections were made through 1-ohm shunts so that the current generated by the galvanic action could be measured in terms of the *IR* drops across the shunts during the course of the test.

Supplementary measurements included the open circuit potentials at the start and the end of the test, the internal resistances of the galvanic cells, and, of course, the weight losses of the several specimens.

Results are given in Table XVI.

It is evident from inspection of these data that the indications from the polarization data were realized in the practical case. The cathodes—titanium and stainless steel—that were polarized most readily had only a slight effect in accelerating corrosion of steel as compared with the more difficultly polarized cathodes, nickel and copper. It is also obvious that the open circuit potential differences between the steel and the other metals, while they indicated correctly the direction of the galvanic action, provided no measure of the magnitude of the galvanic effects in the closed circuits. For example, the galvanic effect of copper was about 14 times that of the stainless steel, although the open circuit potential difference in its couple with steel was over 100 mv less than that of the steel-stainless steel couple. Furthermore, the feeble potential difference between the two pieces of steel was associated with as much galvanic action as was the couple with stainless steel where the open circuit potential difference was from 40 to 50 times as great.

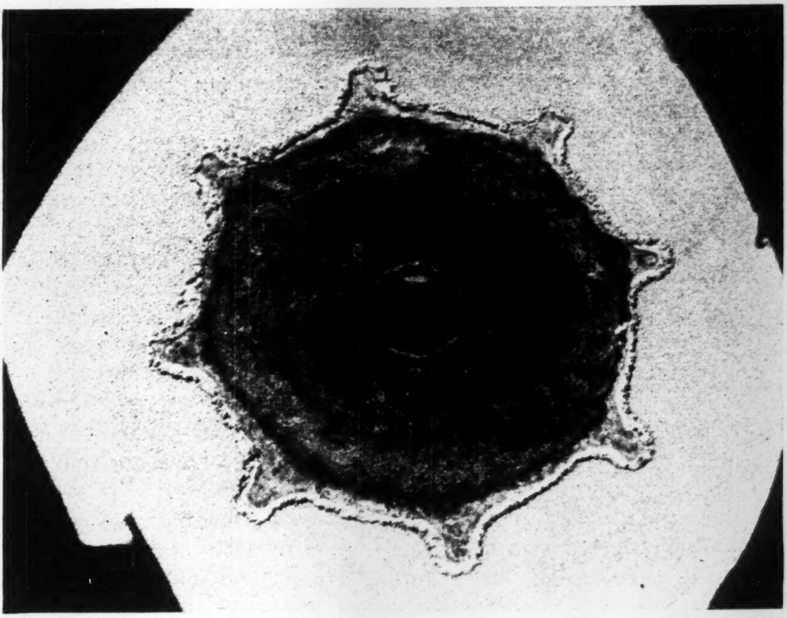


Fig. 45.—Steel Core in Magnesium Casting 9 yr (Marine Atmosphere).

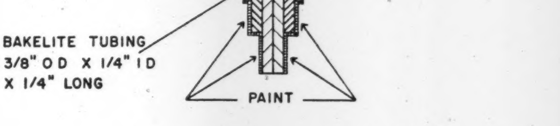

METAL DISKS
1-1/2" O D X 13/32" I D X 1/16" THICK
BAKELITE WASHER
1" O D X 3/8" I D X 1/16" THICK
STANDARD BRASS WASHER
TO FIT # 14-24 SCREW
# 14-24 X 1-1/2" LONG
R.H.B.M.S. WITH BRASS
HEX NUTS
BAKELITE TUBING
3/8" O D X 1/4" I D
X 1/4" LONG
PAINT

Fig. 46.—Atmospheric Galvanic Couple Test Specimen.

Before attempting to use the results of this particular test to predict the relative behaviors of the several metals in galvanic couples in other environments, or in sea water under other conditions, it is necessary to realize that the polarization curves shown and their effects demonstrated by the experiment described are peculiar to the conditions that prevailed in these tests. The same relative behavior may not, and probably will not, prevail under other circumstances. For example, in quiet sea water all of the metals polarize more readily and to about the same extents and all have about the same effect in causing a slight acceleration of the corrosion of steel. Furthermore, the slopes of the polarization curves all become less steep as potentials higher than that of steel are approached, and the effects of the several metals on the corrosion of metals like zinc—less noble than steel—are more nearly alike.

However, this simple example illustrates rather dramatically how important the polarization characteristics of metals can be, not only in connection with their behavior in galvanic couples but also with respect to the reactions that occur in the progress of what we call normal corrosion. It also should serve—we hope—as a stimulus to more intensive study of polarization as a key to our better understanding of why and how metals and alloys corrode as they do. We can look forward to the appearance, someday, of an encyclopedia of polarization data which will provide a basis for the more ready interpretation of corrosion phenomena and the more certain prediction of corrosion behavior.

ATMOSPHERIC GALVANIC CORROSION TESTS

In considering the occurrence of galvanic corrosion in the atmosphere, it is necessary first to have some idea of the distance from the line of contact through which the galvanic effects can extend. An idea of this is provided by the behavior of an assembly of magnesium cast around a steel hub, as shown in Fig. 45, after exposure for 9 yr in the salt spray atmosphere 80 ft from the ocean at Kure Beach. It will be observed that the galvanic acceleration of the corrosion of the magnesium did not extend more than about $\frac{3}{16}$ in. from its line of contact with the steel and that the galvanic (cathodic) protection of the steel was restricted to a similar distance.

It is evident, therefore, that galvanic corrosion in the atmosphere will be highly localized and that the factor of the area relationships that are so important in galvanic action under conditions of immersion in electrolytes will require less attention in atmospheric galvanic corrosion studies. There will be an exception, of course, in the case of small pores through protective coatings where the area exposed at the base of a pore will always be relatively small as compared with the effective area around the mouth of the pore and along the walls of the pore itself. It may reasonably be assumed that since the conductivity of the electrolyte on one metal surface will initially be about the same as on the other, the ordinary condition will represent a couple in which the effective areas of the dissimilar metals will be approximately equal. Thus, the opportunity for the gross acceleration of corrosion that can occur in liquids when the area of the more noble (cathode) metal is relatively very large need not be anticipated in atmospheric exposure. Increases in the conductivity of the electrolyte through the formation of soluble corrosion products will alter the initial conductivity, but generally in a direction to extend the area of attack rather than to increase its depth.

Another major difference between atmospheric and submerged galvanic corrosion is the greater opportunity in the atmosphere to build up insulating corrosion product films that tend in the course of time to reduce the opportunity for galvanic action. For example, couples involving steel with mechanical joints may become almost completely insulated—as in the case of those between steel and stainless steel in the tests reported by Subcommittee VIII of Committee B-3 (55).

The type of atmospheric galvanic couple originally used by Subcommittee VIII is illustrated by Fig. 46. It will be observed that the idea was to confine the

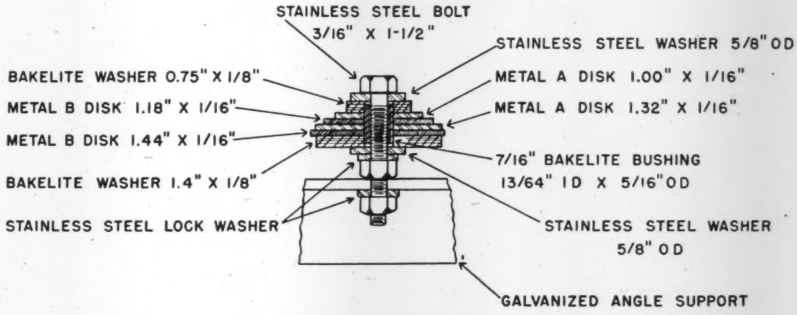
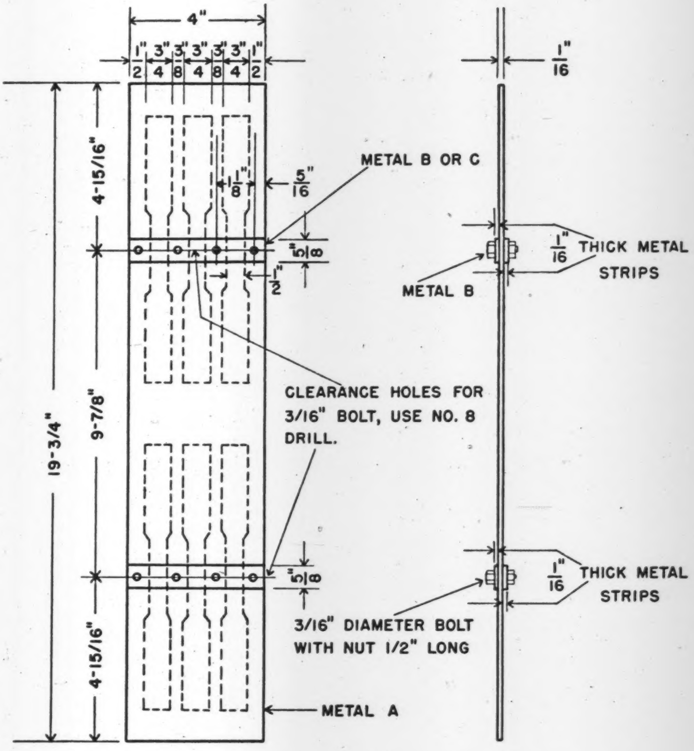

STAINLESS STEEL BOLT
3/16" X 1-1/2"
STAINLESS STEEL WASHER 5/8" OD
BAKELITE WASHER 0.75" X 1/8"
METAL A DISK 1.00" X 1/16"
METAL B DISK 1.18" X 1/16"
METAL A DISK 1.32" X 1/16"
METAL B DISK 1.44" X 1/16"
7/16" BAKELITE BUSHING
13/64" ID X 5/16" OD
BAKELITE WASHER 1.4" X 1/8"
STAINLESS STEEL LOCK WASHER
STAINLESS STEEL WASHER
5/8" OD
GALVANIZED ANGLE SUPPORT

Fig. 47.—Atmospheric Galvanic Couple Test Assembly.


4"
1/2" 3/4" 3/8" 3/4" 3/8" 3/4" 1/2"
1/16"
4-15/16"
1 1/8"
5/16"
METAL B OR C
5/8"
1/2"
1/16" THICK METAL STRIPS
METAL B
19-3/4"
9-7/8"
CLEARANCE HOLES FOR 3/16" BOLT, USE NO. 8 DRILL.
5/8"
1/16" THICK METAL STRIPS
3/16" DIAMETER BOLT WITH NUT 1/2" LONG
4-15/16"
METAL A

TENSION SPECIMENS TO BE CUT AT END OF TESTS

Fig. 48.—Plate and Fastening Type Atmospheric Galvanic Couple Test Specimen.

galvanic effect to the edges of the specimens and to the contacting surfaces to the extent that corrosive liquids might seep into the crevice between the specimens. An effort was made to minimize corrosion outside the area of galvanic action by coating the backs of the specimens with a suitable paint. This introduced a complication in that in interpreting the results it was frequently difficult to decide how much corrosion was due to galvanic action and how much was due to a variable amount of normal corrosion which depended on how soon and to what extent the paint coating had failed to provide protection.

This led to the design of the improved galvanic couple arrangement shown in Fig. 47. In this assembly, each of the two middle specimens has a specimen of the other metal each side of it, and only these two middle specimens are considered in appraising the results. The diameters and thicknesses of the several specimens were chosen so that, when assembled, the area represented by the edge of one specimen and the exposed surface of the specimen on which it rested would be the same in each case. This type of specimen has been quite satisfactory as indicated by the results of the B-3, Subcommittee VIII, studies of atmospheric galvanic corrosion of couples with stainless steels (55).

Another type of specimen that has been very satisfactory is one formed by attaching a strip of one metal to a panel of another one by riveting, bolting, or spot welding, as illustrated by Fig. 48. Here it is possible to check any relative area effects by making either the attached strip or the base panel of the more noble metal. This type of specimen is also well adapted to the assessment of damage by conducting tension tests on specimens that can be cut from the assembly after exposure. The area of greatest possible damage is located in the reduced section of the test piece.

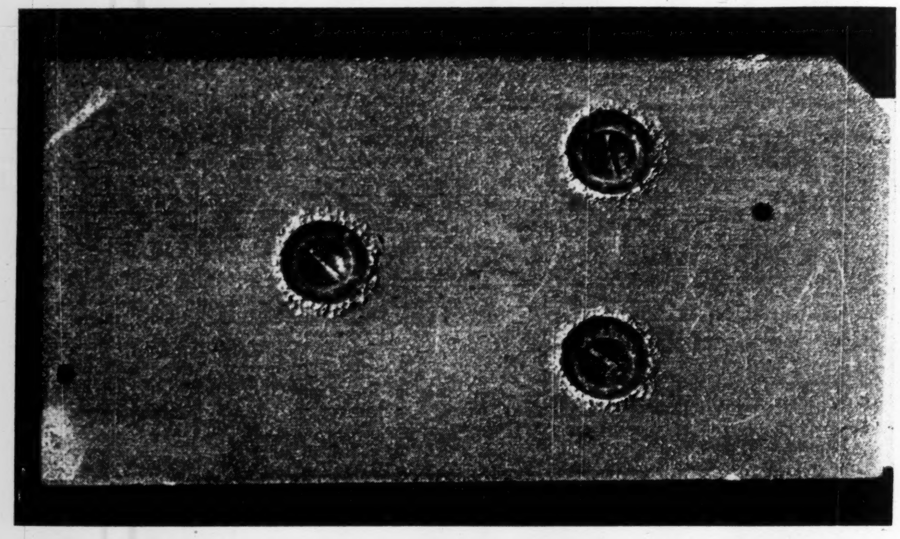


FIG. 49.—Specimen for Studying Galvanic Action Around Fastenings.

A simple modification of this type of specimen is to lap one material over another with the laps usually being horizontal. Usually, also, the lap faces up to permit maximum accumulation and retention of liquid at the line of contact. The relative positions of the dissimilar metals may be changed from top to bottom so as to permit observations of secondary effects of corrosion products that may drain from one material to another and which may be more damaging than the straightforward galvanic effects. In such assemblies, also, there may be effects involving consumption of corrosive constituents of the electrolyte as it passes over one metal before reach-

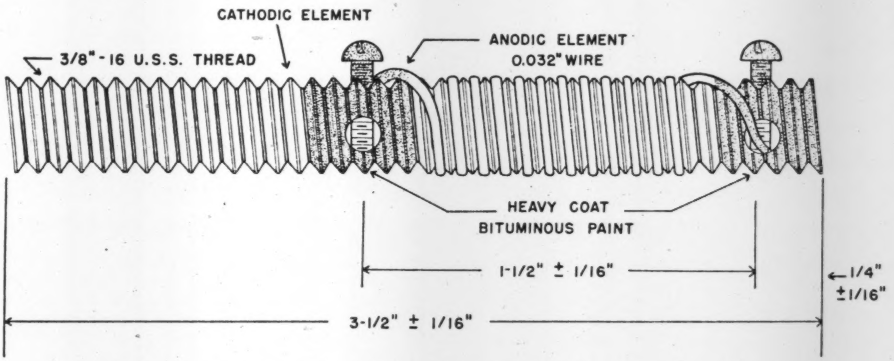

CATHODIC ELEMENT
3/8"-16 U.S.S. THREAD
ANODIC ELEMENT
0.032" WIRE
HEAVY COAT
BITUMINOUS PAINT
1-1/2" ± 1/16"
1/4"
±1/16"
3-1/2" ± 1/16"

Fig. 50.—Bolt and Wire Type Atmospheric Galvanic Couple Test Specimen.

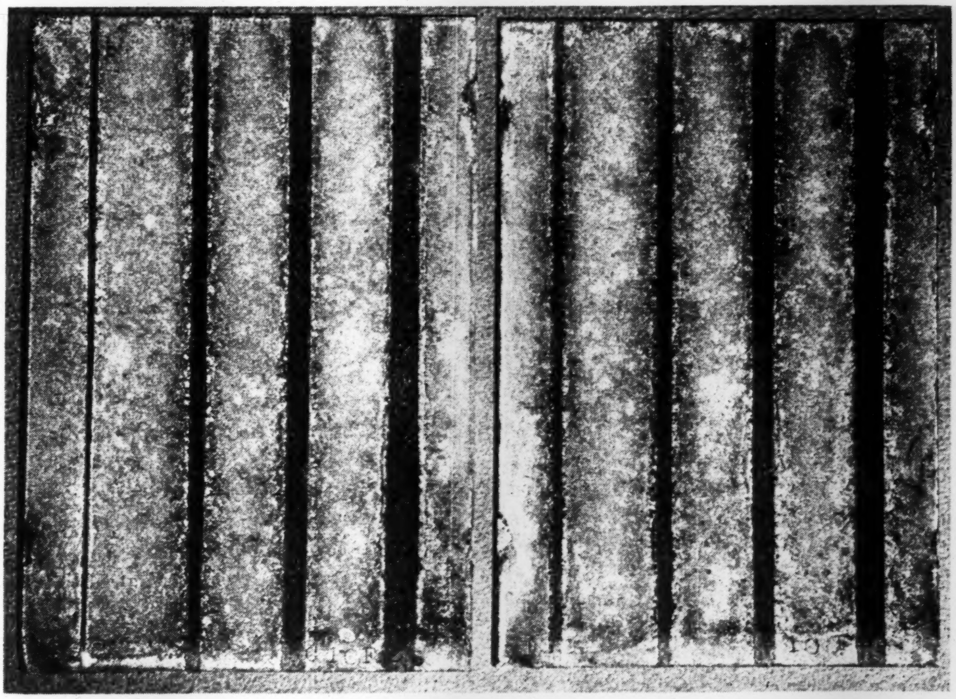


Fig. 51.—Specimens for Studying Galvanic Corrosion Along Bare Strips in Plated Coatings.

ing the other. This will be influenced by the area of the metal that enters into this reaction.

Sometimes the interest is in the galvanic behavior of materials used for fastenings. In this case, the procedure is simply to make some fastening assemblies, such as illustrated in Fig. 49 where the material used for the rivet was one that caused considerable galvanic corrosion of the base panel. This type of assembly also can be made to yield specimens for tension tests after exposure.

A type of assembly calculated to favor a maximum of galvanic action was developed by the Bell Telephone Laboratories as illustrated in Fig. 50. Here the less noble metal is in the form of a wire wound in the grooves of a threaded specimen of the more noble metal and held in good electrical contact with it by set screws covered with a protective coating. This assembly favors accumulation of corrosive liquids around the wire in the thread grooves. Corrosive damage is also favored by the high ratio of surface to mass in the wire specimen. Visual examination is often sufficient to determine the speed and extent of galvanic action. This may be supplemented by weight loss determination or tension tests on the wires as desired. This type of specimen is also being employed by Subcommittee VIII of Committee B-3 in its studies of the atmospheric galvanic corrosion of magnesium.

To observe the extent and through what distance a metallic coating may accelerate or reduce the corrosion of a basis metal, Subcommittee II of Committee B-8 has used specimens of the type shown in Fig. 51. Here strips of varying width are left bare on a basis metal otherwise coated with some other metal—in this case lead electrodeposited on steel. With electrodeposited coatings, the bare strips can be formed simply by applying stop-off lacquer prior to plating. The extent of corrosion on the bare strips as compared with that on a completely bare or completely coated specimen will provide a measure of galvanic action (increase or decrease in corrosion) and the distance to which it may extend from the edges of the bare strips.

This and other tests that may be made with porous metallic coatings will provide data that will indicate the direction of galvanic effects in different atmospheres and will permit the setting up of galvanic series in which the relative positions of different metals will vary with the type of atmosphere—whether rural, marine, or industrial.

SALT SPRAY TESTS

The salt spray or, more correctly, the salt fog test has been somewhat of a problem child within the Society as well as elsewhere since it was first introduced by J. A. Capp (56) in 1914. This situation has been due in part to some inherent limitations of the test itself, but even more to the misuse of the test (57) for purposes for which it never was intended and for which it is fundamentally unsuited.

Further complications have been introduced by the fact that the results secured are influenced to a considerable extent by conditions of the test which either have not been standardized or cannot be standardized. These include such factors as the position of the test pieces, the fog density, the size of the fog particles, the temperature of the chamber, the pressure at which the brine is sprayed and the constancy of this pressure, the concentration of salt and other chemicals in the brine, the pH of the brine, and the size and shape of the fog chamber. There are, also, frequently encountered difficulties in establishing satisfactory criteria of performance.

This situation was improved considerably by the action of the Society through Subcommittee III of Committee B-3 in setting up standards covering such features as brine concentration, temperature and fog density as covered by Tentative Method B 117 (58).

Mr. Capp's original idea was to provide in an inland laboratory a reasonable duplication of the corrosive environment that might be encountered near the ocean and to maintain such corrosive conditions continuously so as to achieve in a relatively short time as much damage as would occur over an extended period of exposure to the natural conditions. His original aim was to determine the quality or protective value of metallic coatings. However, the test has since been extended to such purposes as testing organic finishes (paints), protective oils and greases, and even solid metals and alloys, for example, stainless steels for which it is not well suited (59) either to measure the corrosion-resisting qualities of these steels or for detecting surface contamination by iron, the effects of which may be masked by the natural rusting of the stainless steel in the test.

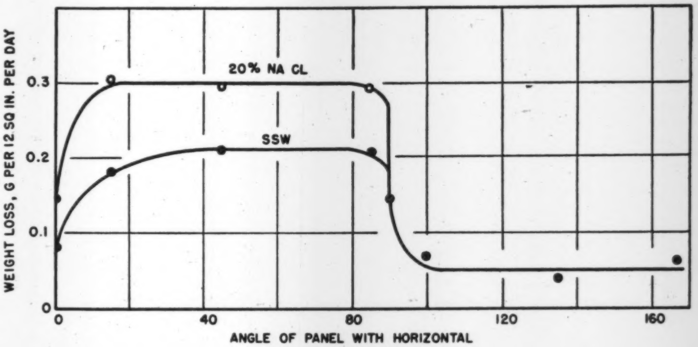


Fig. 52.—Effect of Specimen Angle on Corrosion in Salt Spray Cabinets Using 20 per cent Sodium Chloride and Synthetic Sea Water.

The salt spray test is of little value for evaluating the relative merits of different metals and alloys but has been useful in comparing different modifications of materials of the same general class as, for example, aluminum alloys for which the test has yielded generally satisfactory results in terms of relative performance under natural conditions of exposure. For testing aluminum alloys, some investigators prefer an intermittent over a continuous spray test (60).

A salt spray box is simply a device by which specimens placed in it can be subjected to the corrosive action of a film of the brine that settles out of the fog or mist created by the jets through which the brine is forced into the chamber. This brine film can attack a metal, a coating on a metal, or the metal itself where it may be exposed through a coating. The relation between what happens in the salt spray box and what happens under natural conditions of exposure is influenced by the following features:

Settlement of Brine Particles:

The brine particles settle substantially vertically so that surfaces exposed at different angles will encounter differenta mounts of the corrosive brine. As demonstrated by May and Alexander (61), practically all of the corrosion occurs on the upper surfaces of specimens upon which the brine particles can settle. This is illustrated by their data shown in Fig. 52. Small variations in angle of exposure around 90 deg may have a great effect on the amount of surface exposed to attack with corresponding great variations on the extent of corrosion of duplicate specimens. It is desirable, therefore, to expose flat panels at an angle from the horizontal of from 30 to 80 deg where small variations in angle from specimen to specimen will have a negligible effect on corrosion. Under such circumstances, practically all the corrosion will occur on the upper surfaces which will, therefore, be the only significant ones in appraising the results of the test. It may be recalled in passing that this distribution of corrosion on inclined steel specimens in a salt spray box is quite different from what develops in a natural atmosphere where more corrosion occurs on the groundward than on the skyward surfaces.

Distribution of Fog in Test Chamber:

The distribution of the fog through the chamber and the rate at which it settles may not be uniform so that specimens located in one place may come into contact with more brine than those resting somewhere else. This naturally will affect the reproducibility of results in any one chamber and between chambers of different size, shape, and nozzle arrangement.

Effect of Spraying Conditions:

The nature of the fog with respect to particle size and rate of settling are influenced by the characteristics of the spray nozzles, the pressure at which the brine enters the nozzle, and the constancy of this pressure (62).

Corrosive Characteristics of Brines:

The corrosive characteristics of the brine films that settle on surfaces in a salt spray chamber are peculiar to themselves and cannot be expected to duplicate the corrosive nature of films that may wet surfaces exposed to natural environments. Similarly, different brines will have different corrosive characteristics and cannot be expected to give the same results. A weak brine may be more corrosive to one material, while a strong brine may be more aggressive towards another. Since neither brine can be expected to match the corrosivity of some different natural environment towards materials in general, there is no strong reason to prefer one concentration of brine over another except for the sake of favoring uniformity of results of tests by different investigators. This reference to "uniformity" of results does not make any distinction between results that are uniformly consistent and those that are uniformly inconsistent in terms of behavior under some different natural conditions of exposure. Here uniformity is prized only for its own sake.

There seems to be no real justification for going to the trouble of making up a complicated synthetic sea water solution for use in salt spray boxes. A possible improvement in this one factor cannot be expected to have any great effect on the over-all consistency of results relalive to performance under natural service conditions that differ in so many ways from those that can be realized in a salt spray test chamber.

When it is known that a certain composition of brine is most corrosive towards a particular material, and the object of the test is to achieve maximum destruction in minimum time, this concentration of brine is indicated in preference to some other one. But it must be remembered that this composition of brine may be less corrosive than another one towards some other material and that no single brine composition can be used to rate materials in any general order of corrodibility that will apply to other environments, including even marine atmospheres.

Some of these points are illustrated by data provided by May and Alexander (61) reproduced in Figs. 53 and 54. One of the most surprising results of these studies was the negligible acceleration of corrosion of iron in the standard 20 per cent salt spray as compared with natural exposure 80 ft from the ocean. On the other hand, both the 3 per cent

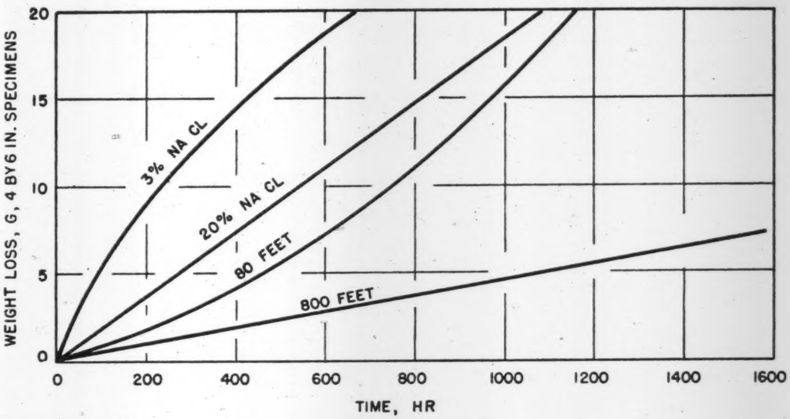


Fig. 53.—Comparative Tests on Open-Hearth Iron in Sprays of 3 per cent and 20 per cent Sodium Chloride and in the Marine Atmosphere 80 ft and 800 ft From the Ocean at Kure Beach, N. C.

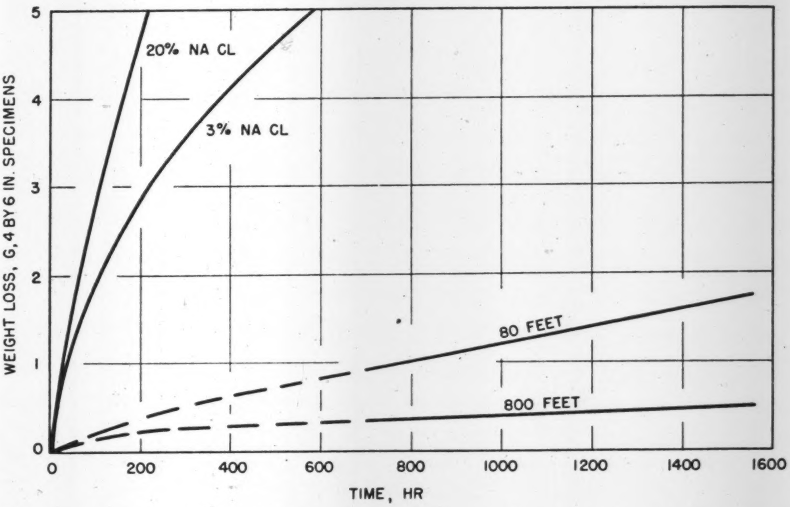


Fig. 54.—Comparative Tests on Zinc in Sprays of 3 per cent and 20 per cent Sodium Chloride and in the Marine Atmosphere 80 ft and 800 ft From the Ocean at Kure Beach, N. C.

and 20 per cent brine sprays achieved considerable acceleration in the corrosion of zinc, with the 20 per cent brine being the more aggressive.

The big difference in corrosion 80 ft from the ocean as compared with 800 ft fron the ocean—a ratio of about 3 to 1 for steel and 5 to 1 for zinc—illustrates the impossibility of achieving any precise definition of what is meant by a "marine atmosphere" as a basis of comparison with results of tests of a variety of materials in other natural or artificial environments. Any notion or statement that so many hours exposure in a salt spray chamber is equivalent in general to so many days, weeks, months, or years in a natural environment (including marine environments) is obviously nonsensical.

Effects of Corrosion Products:

As has been pointed out before, it is necessary in appraising the corrosion-resisting qualities of metals to make a proper distinction between the corrodibility of the metal and the protective qualities of its corrosion products. The corrosion products that are formed in a salt spray chamber may have quite different chemical and physical characteristics as compared with those formed under natural conditions of exposure and will, therefore, have quite different influence on the progress of corrosion. (For example, see Fig. 29 which compares the behavior of the specimens from the rimmed steel ingot in salt spray tests and in the natural salt atmosphere.) This will account for many discrepancies between results of salt spray tests and performance under natural conditions.

Electrolytic Effects:

The conductivity of the brine film electrolyte that wets the surface of a specimen in a salt spray chamber may be much higher than that of the films of rain or moisture that form on materials exposed in natural atmospheres and much lower than that of the electrolyte that may surround materials when they are immersed in some corrosive liquid.

Where combinations of dissimilar metals are involved, as in the case of one metal coated with another, the galvanic relationship between the dissimilar metals in the electrolyte in the salt spray chamber may be quite different from that in some other natural environment.

These factors influence the magnitude, the distribution, and even the direction of the galvanic effects that may be involved.

Anodic Coatings:

In the particular case of metallic coatings, a salt spray test will not be effective in disclosing the size or number of pores in a coating that is anodic to the basis metal (such as zinc on iron) since galvanic currents will flow from the coating to the exposed basis metal at a sufficiently high current density to provide complete cathodic protection and thus prevent any visible corrosion of the basis metal.

As indicated by the data in Fig. 55, the attack on some anodic coating materials, such as zinc, in a salt spray test is roughly directly proportional to time and proceeds at a fairly high rate. Thus, performance in the salt spray test may serve as a convenient measure of the thickness of a coating. However, it appears to have little or no advantage over more direct and even faster methods of measuring coating thickness.

Some have claimed that spot measurements of coating thickness do not provide a good picture of the uniformity of the thickness of the coating from point to point and have suggested that the salt spray test has the advantage that the whole surface is subjected to inspection by this test. However, this latter advantage becomes more apparent than real when it is recalled that the deposition of the brine fog particles varies through very wide limits depending on the angle of exposure of the specimen (see Fig. 52). This will be particularly significant when the specimen is in the form of some manufactured article of complicated shape having surfaces disposed at a variety of angles.

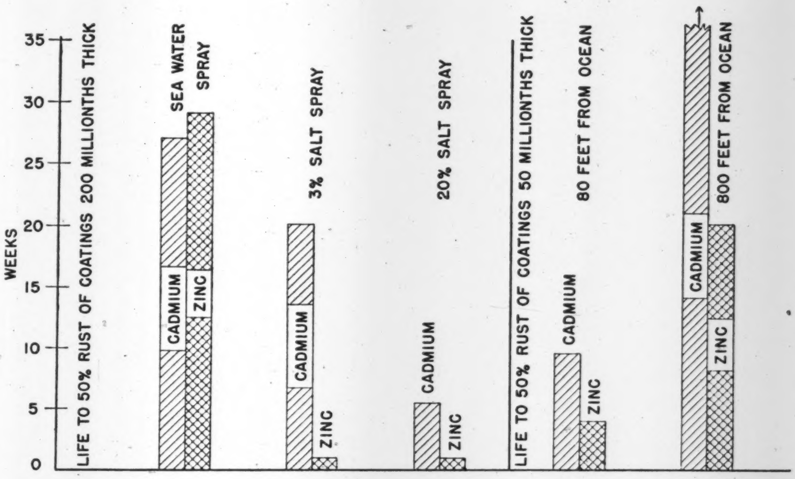


Fig. 55.—Comparison of Cadmium and Zinc Plating in Salt Spray Tests and Marine Atmospheres.

Consequently, the life of a coating in a salt spray test may be determined:

(*a*) By its thickness.

(*b*) By the amount of corrosive brine it encounters.

Thus, differences in the time required to destroy a coating at different points on a test specimen may indicate differences in the extent of settling of the corrosive brine particles on different surfaces fully as much as differences in coating thickness from point to point. Consequently, a salt spray test cannot be considered to be a reliable means of measuring the thickness and uniformity of a coating, especially on specimens or articles of complicated shape.

Immersion of a coated specimen in an agitated solution that will reach and corrode the coating on all surfaces at the same rate would be much more reliable than a salt spray test as a means of using destructive corrosion for inspecting a coating with reference to its thickness from point to point.

Comparison of Zinc and Cadmium on Steel:

The failure of salt spray tests to make a proper distinction between the protective value of zinc and cadmium coatings on steel has been cited frequently (63). Probably the principal reason for this discrepancy is that under the conditions that exist within a salt spray chamber cadmium corrodes substantially less than zinc (especially in the 3 per cent salt brine spray) whereas in other environments, such as industrial and rural atmospheres cadmium corrodes as fast, or faster, than zinc.

The situation with respect to marine atmospheres has been somewhat obscure, but results of recent tests on specimens provided through the courtesy of A. Mendizza of the Bell Telephone Laboratories are throwing considerable light on this subject. These results will be reported in detail in some future paper, but, as illustrated by Fig. 55, it already appears that at least with thin coatings (under 0.0005 in.) the superiority of cadmium indicated by the salt spray tests extends as well to natural exposure in marine environments. It may be that the building up of protective corrosion product films on zinc under natural conditions of marine exposure will improve its protective value relative to cadmium and that the indicated degree of superiority of thin coatings of cadmium will not extend to thick coatings, for example 0.002 in. and heavier.

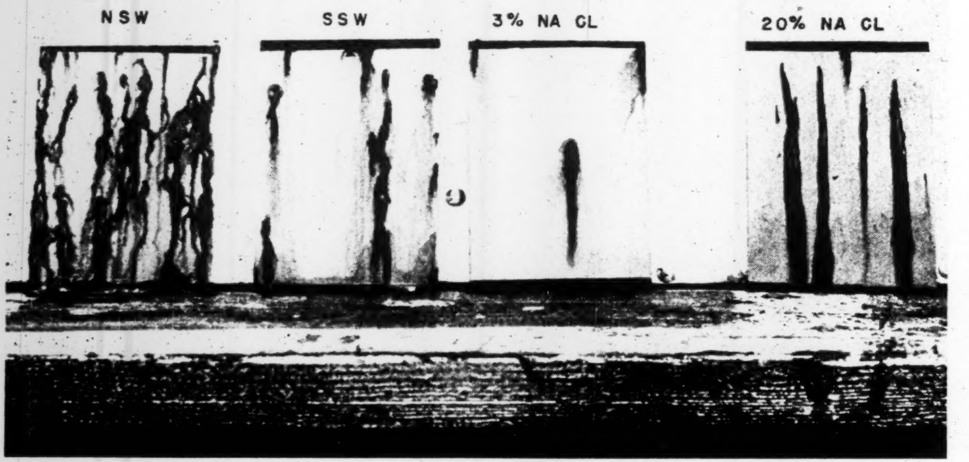


FIG. 56.—Development of Rust Spots in Nickel-Plated Steel Specimens in Salt Spray Tests Left to Right: Natural Sea Water Spray, Synthetic Sea Water, 3 per cent NaCl, 20 per cent NaCl.

Cathodic Coatings:

In the case of coatings that are cathodic to the basis metal (such as nickel, copper, and chromium on steel) the tendency will be for the galvanic effect to accelerate corrosion at any spots where the steel is originally exposed and thus expedite the disclosure of their presence. However, the current flowing to the coating around such areas of active corrosion of the basis metal will provide some cathodic protection of the coating and thus restrain the rate of thinning of the coating and the development of pits through it in the vicinity of the first rust spots to develop. The distribution of this cathodic protection will be influenced by the conductivity of the film of electrolyte on the surface. Where this is high, as in a salt spray chamber, a few bare spots may serve to protect the coating over the rest of the test specimen and thus confine attack to the few points where it starts first. Where the conductivity of the surface films is much lower, as in a natural atmosphere, the protective cathodic currents will be feeble and restricted to the immediate vicinity of bare spots so that the presence of one may not prevent the development of another one near it as a result of corrosion of the coating. Thus, the distribution and number of points of attack and the deterioration of the coating itself on a cathodically coated metal under natural conditions of exposure may be quite different from that indicated by a test in a salt spray box.

This is illustrated by comparative tests on duplicate steel specimens carrying a thin (0.001 in.) coating of nickel exposed in a 3 per cent salt spray box and in the natural marine atmosphere at Kure Beach. As will be seen in Fig. 56. the attack on the specimens in the spray box was confined to the few points at which it started and which apparently represented bare spots originally present. However, in the natural atmosphere, the failure of the coating took the form of the development of a multitude of tiny pits distributed at random. Apparently the relatively poor conductivity of the films of electrolyte that formed in the natural atmosphere restricted the area of the cathodic coating that was able to affect the corrosion of any existing bare spots so that the attack on these was not accelerated greatly. Likewise, there was not enough flow of galvanic current to the coating itself to prevent it from becoming penetrated by corrosion at many points with the consequent appearance of widespread attack visible in Fig. 57.

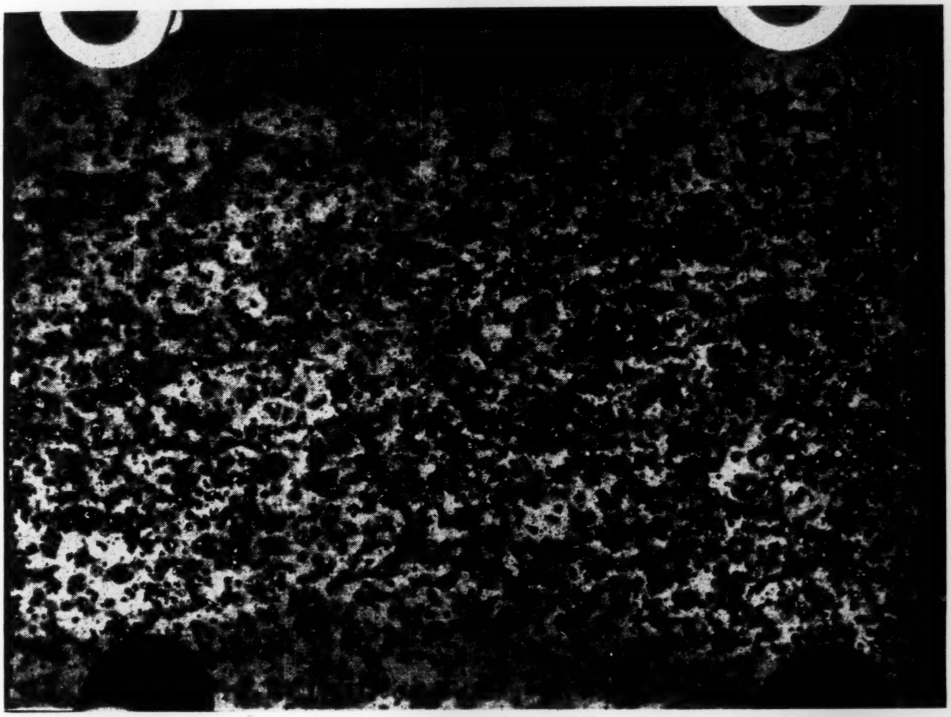
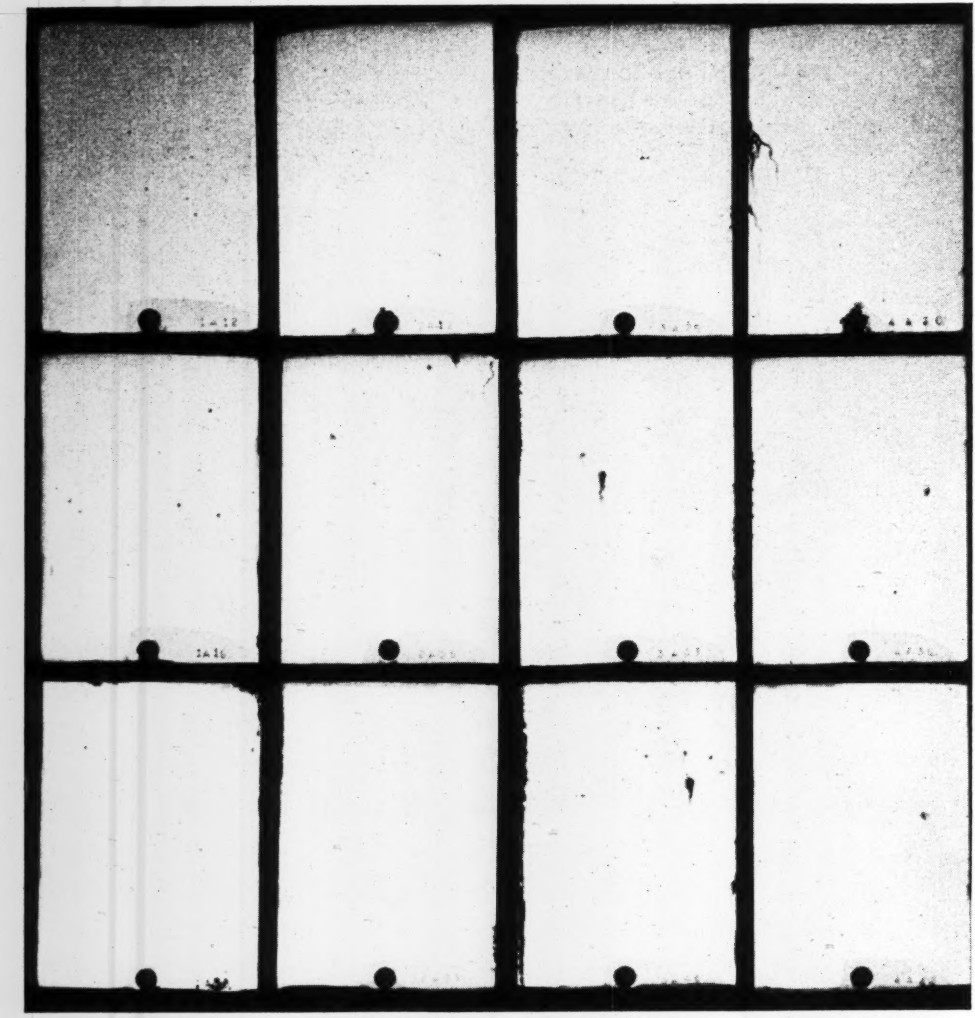


Fig. 57.—Pattern of Rust Development on Nickel-Plated Specimens Exposed to Natural Marine Atmosphere.

It follows, then, that where cathodic coatings are concerned a salt spray test will be most useful in disclosing the presence of existing bare spots but it cannot be expected to predict the occurrence of bare spots and rusting that may be the result of attack of the coating by the natural environment in which it will be used and which may corrode the coating much differently from what occurs in a salt spray box.



0.001 in. Ni

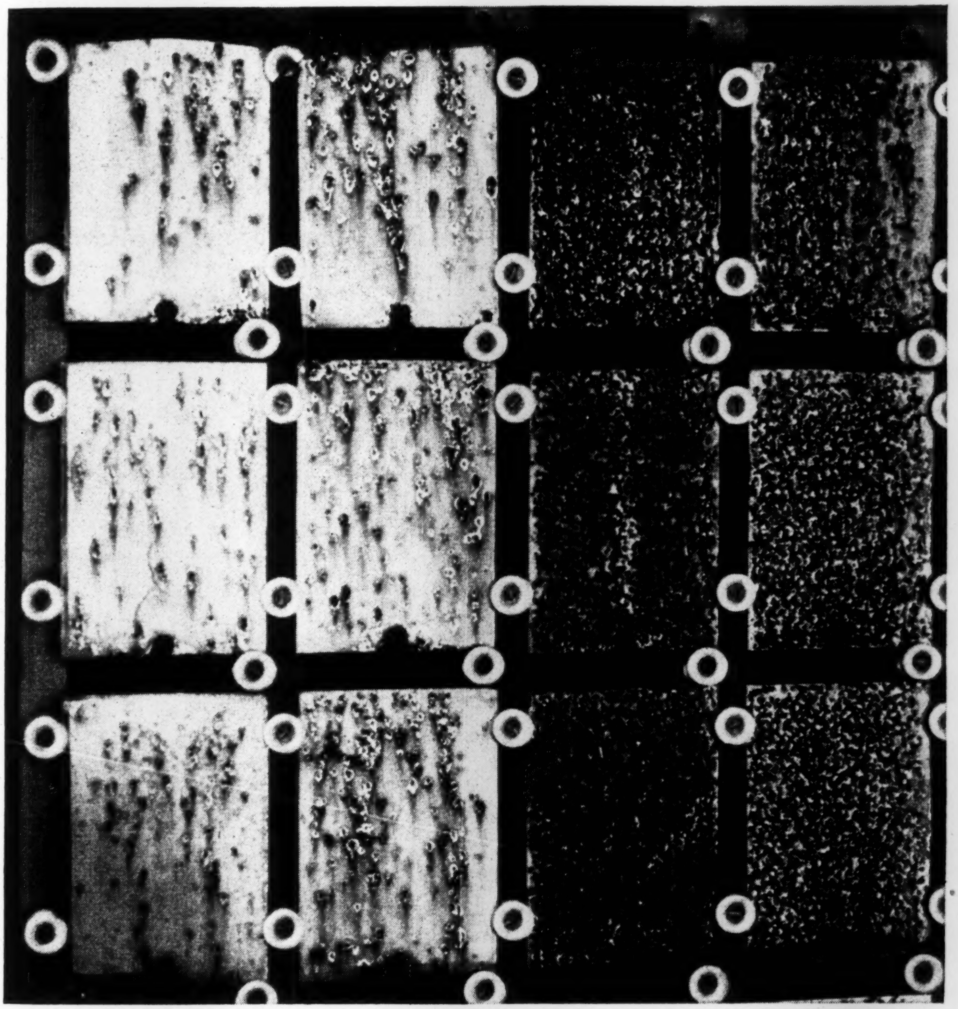
0.0001 in.Cu + 0.0009 in. Ni

0.0005 in. Cu + 0.0005 in. Ni

0.0005 in. Cu Buffed + 0.0005 in. Ni

FIG. 58.—Appearance of Nickel and Nickel plus Copper Plated Specimens After Salt Spray Test.

This latter point is illustrated well by the comparison of the behavior of chromium on nickel plating and chromium on a combination of nickel and copper plating when exposed in a salt spray box and when exposed in a marine atmosphere by Subcommittee II of ASTM Committee B-8 (64). It will be seen from the photographs in Figs. 58 and 59 that the salt spray test failed to disclose the big difference in behavior of the all nickel as compared with the nickel plus copper coatings with respect to the development of rust spots in the natural marine atmosphere.



0.001 in. Ni

0.0001 in. Cu + 0.0009 in. Ni

0.0005 in. Cu + 0.0005 in. Ni

0.0005 in. Cu Buffed + 0.0005 in Ni

Fig. 59.—Appearance of Nickel and Nickel plus Copper Plated Specimens After Exposure to Natural Marine Atmosphere.

Acid Sprays:

The brines used in standard salt spray tests are maintained nearly neutral (pH 6.5 to 7.2). However, such brine sprays do not reproduce the kind of blistering attack that has been observed frequently on chromium, nickel, copper-plated zinc-base die castings such as are used for atuomobile hardware and exposed to acid industrial atmospheres sometimes contaminated with salt used for deicing roads. To guide the development of plating resistant to this sort of deterioration, it has been helpful to devise an acid spray (65) using acetic acid as a convenient well-buffered source of the desired acidity.

It seems likely that other modifications of the standard sprays will be developed as required in the course of future research in the improvements of protective coatings.

These are some desirable qualities of a coating that generally increase with its thickness. For example, the number of original pores in a nickel coating decreases as the thickness of the coating increases. Consequently, the salt spray test which will disclose the presence of original pores provides a rough means of inspection for thickness of nickel coatings. However, the non-uniform settling of salt fog particles on different surfaces greatly reduces the usefulness of the salt spray test for any precise general inspection of large surfaces, especially on complicated shapes.

The salt spray test may also be used for studying the protective qualities of non-metallic coatings, such as paints, oils and the like. Here, though, the prime interest is in the continuity and degradation of the coating, the test environment is chosen on the basis of how it affects the metal that is coated rather than the coating itself. The relation between behavior of such coatings in the salt spray box and their behavior in a natural environment seems to be even more obscure and uncertain than in the case of bare metals and metallic coatings as already discussed. It also is complicated further by the natural effects of sunlight, thermal shock, etc., which appear to affect the deterioration of organic coatings more than they do corrosion of metals and by effects of the severe washing effects of heavy wind-blown rains which may be particularly important in the case of tests of slushing oils and greases which never encounter some of these effects in a salt spray box.

Efforts have been made to take care of some of these extra factors by installing ultra-violet lamps in salt spray chambers (66) and by operating the boxes at higher than normal temperatures.

Alexander and May (67) compared the deteriorating effects of different brine sprays on painted steel, aluminum, and magnesium and discovered that the 3 per cent and 20 per cent sodium chloride brine sprays were more destructive than either natural or synthetic sea water spray and that especially in the case of painted steel, the 3 per cent brine spray was more drastic than the 20 per cent brine spray. But, here again, the many other factors that influence behavior in service as compared with behavior in a salt spray test make it seem unlikely that the over-all consistency between the results of a salt spray test and those secured under natural conditions will be improved significantly by the use of one spray solution in preference to another. It is suggested, then, that since the principal object of making such a test is to get some sort of result quickly, the brine that produces the greatest effect in the shortest time (for example, a 3 per cent brine on painted steel) might reasonably be used. The trouble of making up a complicated synthetic sea water brine can hardly be justified for general use.

While the present author has not had much experience with the use of salt spray boxes for testing organic coatings, he hazards the opinion that the following remarks based primarily on experience with metals and metallic coatings will extend to the testing of organic coatings as well.

In spite of the limitations that have been described, and in part because of their recognition by competent investigators, the salt spray test has been a useful tool (particularly for inspection) in the hands of experienced operators. Its utilization has been paralleled by a steady improvement in the protective value of coatings—achieved generally in the case of metallic coatings simply by making them thicker. It seems likely, however, that the extent and speed of further improvements will be increased less by further dependence on salt spray or similar tests than by the development and use of means of evaluating, both individually and combined, those qualities and characteristics of coatings upon which their protective value depends.

Humidity Tests:

The humidity or water vapor condensation tests are basically quite similar to salt spray tests. The principal difference is that the film of liquid that covers the surfaces of the test specimens is substantially pure water rather than some sort of brine. In fact, a reasonably satisfactory humidity test may be made simply by spraying pure water instead of a brine in a conventional salt spray cabinet. In this connection, it is interesting to note that Capp (56) used water in his spray test before he used brine.

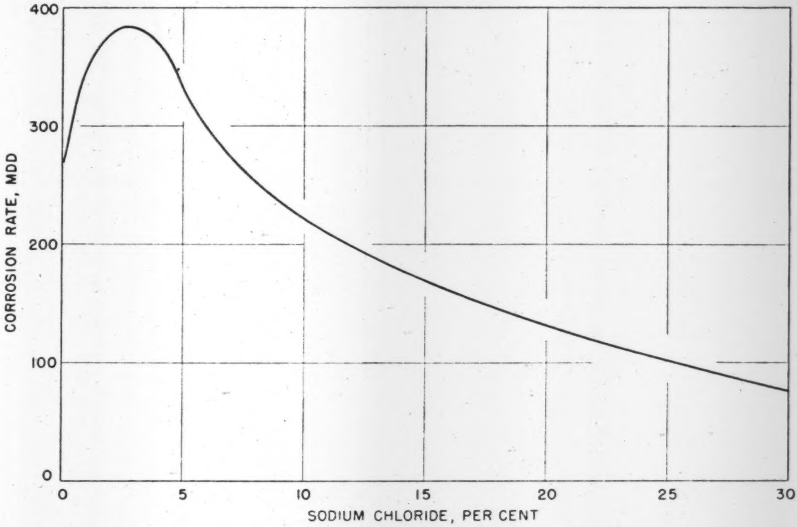


Fig. 60.—Effect of Salt Concentration on Corrosion of Steel in Alternate-Immersion Test at 30 C, 90 per cent Humidity.

Such well-aerated water films are surprisingly corrosive. As a matter of fact, because of the greater solubility of oxygen in water as compared with strong brines, the former may be even more corrosive to iron than the latter. This is illustrated by results of alternate immersion tests conducted by Portevin and Herzog (68) as shown in Fig. 60.

The electrical conductivity of water

films is much lower than that of brine films and is more nearly like that of the rain and moisture films that occur in natural atmospheres. Thus, it may be expected that the distribution of corrosion on humidity cabinet test specimens will be more like what develops under natural exposure conditions than is the case with salt spray tests as already discussed.

Up to now the design and operation of humidity test cabinets has not reached the degree of standardization that will be required before reproducible results can be expected from different laboratories. But, with the attention being given this problem by Committee E-1, it can be expected that this situation will be corrected before long and humidity tests will take their proper place among the tools provided by the Society for measuring and improving the corrosion-resisting qualities of metals and coatings.

LABORATORY TOTAL IMMERSION TESTS

After many preliminary investigations, including "round-robin" corrosion test programs under the auspices of Committee B-3, Subcommittee I, the Society established a recommended procedure for carrying out total immersion corrosion tests in a laboratory. This is covered by Tentative Method B 185 (69).

In the course of the studies sponsored by Committee B-3, W. A. Wesley (70) demonstrated by examples of monel in sulfuric and hydrochloric acids, and steel in sodium chloride, that with the control of temperature, velocity, and aeration that is practical in a laboratory setup

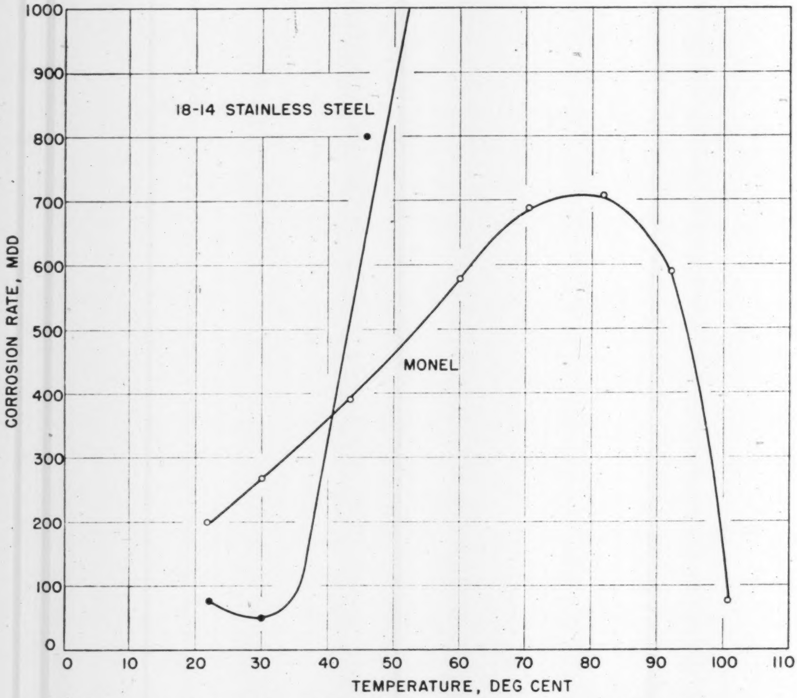
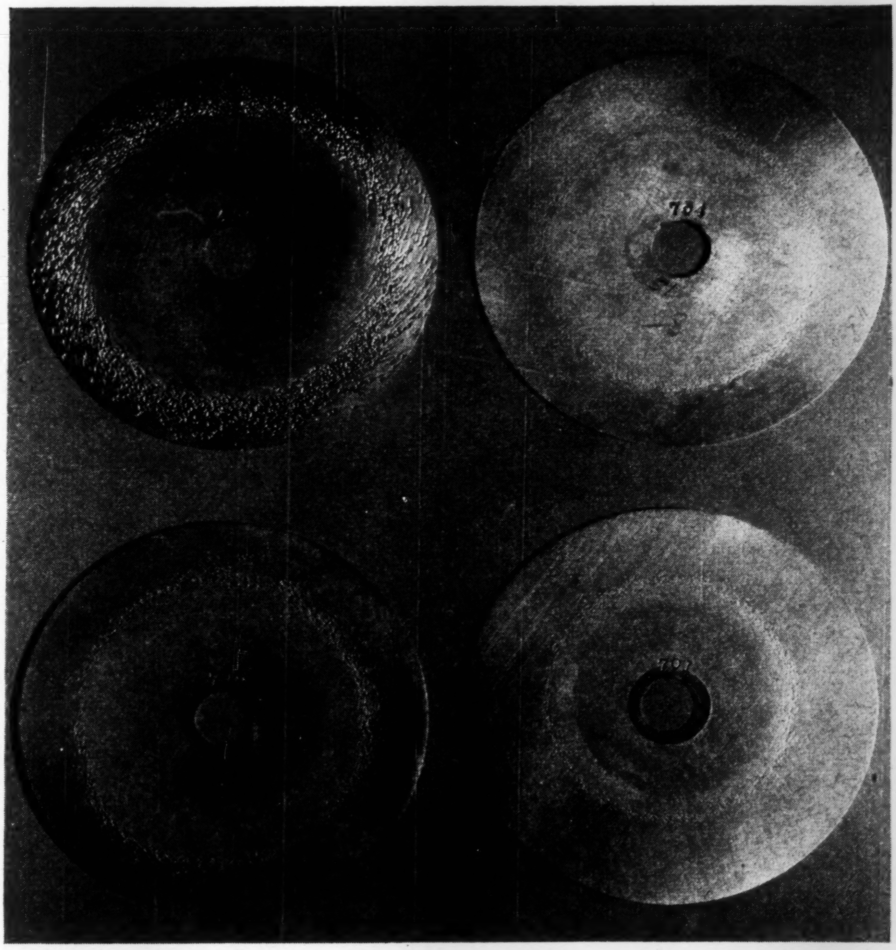


FIG. 61.—Corrosion of Metals in 5 to 6 per cent Sulfuric Acid.

(Air Saturated Acid Solutions; Velocity, 15.5 ft per min.)

and is called for in Method B 185, it is possible to secure satisfactory reproducibility of results not only amongst specimens in a particular test, but from test to test undertaken at different times. It is evident, therefore, that the apparatus used by Wesley provides for proper control of the important factors. This does not mean that this apparatus is the one that must be used but that any mechanism which will permit equally satisfactory control of temperature, aeration and velocity also can be expected to yield results of satisfactory reproducibility.



Admiralty Brass.

90-10 Copper-Nickel, 0.8 per cent Iron.

70-30 Copper-Nickel, 0.5 per cent Iron.

70-30 Copper-Nickel, 0.05 per cent Iron.

Fig. 62.—Disk Specimens From Rotating-Spindle Test in Sea Water.

The recommended procedure provides for control of such important variable factors as temperature, aeration, and velocity without attempting to fix any of these within prescribed limits in the form of "standard" testing conditions. It was recognized, for example, that if the degree of aeration were to be held to a high value, such as air saturation, the corrosion of one class of material (for example, ordinary steel and copper) would thereby be raised to a maximum value, while that of another class (those passivated by oxygen, for example, stainless steels) would be reduced to a minimum value so that no over-all comparison covering a range of conditions of aeration would be provided.

In the case of the control of temperature, the problem becomes complicated by the many ways in which temperature can affect corrosion, such as:

1. Increasing rates of chemical reactions.
2. Reducing the solubility of dissolved gases, including oxygen—this may cause an abrupt increase in corrosion of stainless steels that depend on oxygen for passivity, or an ultimate decrease in corrosion of alloys like monel that do not corrode much in the absence of oxygen. This is illustrated for both cases by the results of tests in sulfuric acid shown in Fig. 61.
3. Changing the concentration of a solution by evaporation.
4. Precipitation of compounds—sometimes as protective scales—as a result of thermal decomposition or changes in solubility.
5. Increasing the solubility of corrosion products that are protective at lower temperatures, for example, lead chloride in hydrochloric acid.
6. Changing the physical (protective) qualities of corrosion products, for example, zinc in water (**71**).

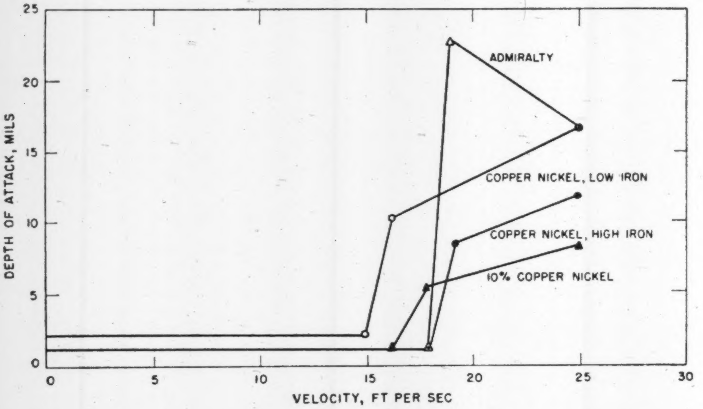


FIG. 63.—Effect of Velocity on Depth of Attack in Sea Water at Kure Beach, N. C. for 60 Days.

It is evident, therefore, that no particular temperature can be considered an ideal one for securing data having broad significance. For arbitrary tests where there is no other basis for preferring one temperature over another, it is generally desirable to use a temperature slightly above average room temperature, for example, 30 C (86 F) because of the greater convenience of holding this temperature by thermostatic control of the input of heat as compared with efforts to control temperature at a lower value by cooling.

The study of effects of velocity is particularly troublesome for the following reasons:

1. It is difficult to achieve precise high velocities because of the tendency of friction films to travel with moving test specimens or to adhere to stationary ones, thus making the estimation of the true relative velocity very uncertain.
2. Variations in relative velocity from point to point may introduce complicating effects of differential aeration or metal ion concentration cells which affect different metals in different ways.
3. The erosive effects associated with high

velocities may remove or interfere with the formation of protective films which often are major factors that determine not only the rate of corrosion, but whether it will occur at all. This is illustrated by a method of studying effects of velocity on corrosion of metals by sea water which involves the use of horizontal disks mounted on a vertical shaft and which are whirled through the water at high speed. As these specimens rotate there is a natural variation in velocity from center to periphery depending on the radius of rotation. The effects under discussion are illustrated by the appearances of specimens from such a test shown in Fig. 62. The numerical data are shown in Fig. 63 in which the measured decrease in thickness as a result of corrosion is plotted against the calculated velocities from point to point between the center and the periphery. The abrupt increase in attack that follows the removal of protective corrosion products is clearly evident. It will be seen that:

(*a*) Up to the velocity at which the several alloys were able to retain their protective films there was no appreciable difference in the extent of attack.

(*b*) Beyond the velocity at which any of the alloys were able to hold their protective corrosion products there were small differences in corrosion rates.

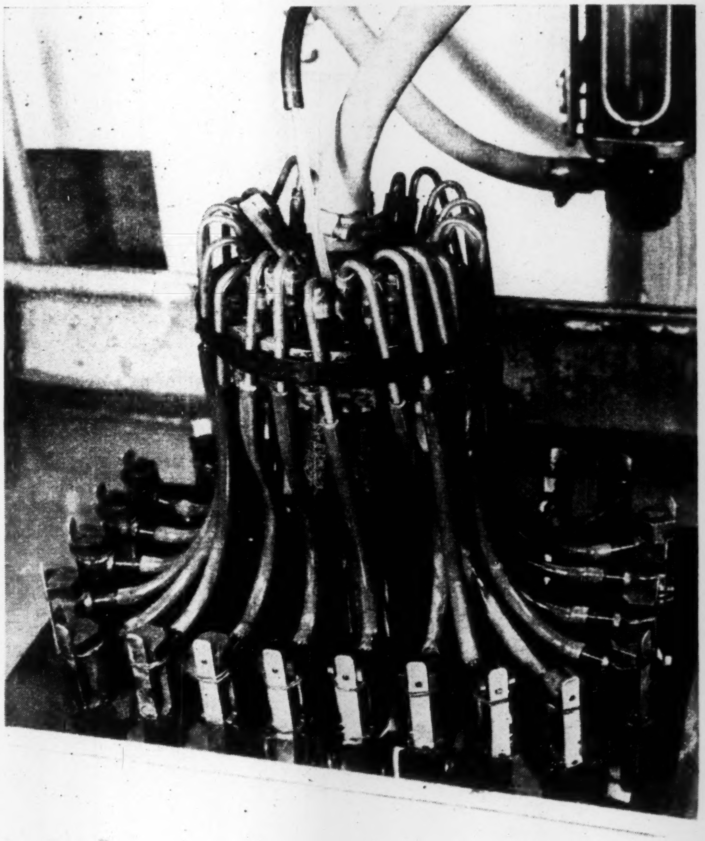
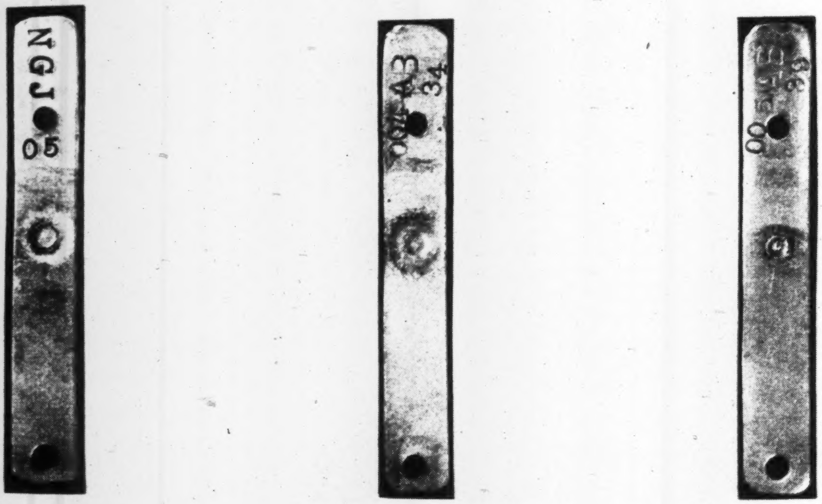


Fig. 64.—Jet Impingement Test Apparatus as Used by British Non-Ferrous Metals Research Association.

(*c*) The important information to be secured from tests of this sort is not only the rate at which attack occurs below or above some critical velocity, but rather how different metals compare with respect to the critical velocity that marks the boundary between the maintenance and loss of protective films. In referring to such a "critical velocity" it must be remembered that its value will be influenced by the way in which it is determined. Because of the effect of different velocities from point to point, as on a rotating specimen, the apparent "critical velocity" on such a specimen may be quite different from what will be indicated by tests in which the velocity is more uniform from point to point, as in the case of a corrosive liquid passing over a stationary specimen—as through a test specimen in the form of a pipe or with a device such as illustrated in Fig. 40. Consequently, there can be no such thing as a critical velocity that will apply to all circumstances of use. The establishment of critical velocities by a particular method of test will afford only a qualitative comparison amongst a number of materials as to their relative abilities to resist the destructive effects of high velocities.



Arsenical Aluminum Brass Admiralty Brass Aluminum Brass

FIG. 65.—Specimens From British Non-Ferrous Metals Research Association Jet Test Apparatus. Exposed 9-1-50 to 10-1-50.

Various devices are used to whirl specimens through testing solutions. Some of these employ disks upon which suitable specimens can be mounted as described, for example, by LaQue and Stewart (72) in connection with tests in sea water. Such rotating disk specimen holders have been used also by British investigators (73) for carrying out what are called "accelerated" tests of paint coatings for underwater service.

Another apparatus that has been employed for testing effects of velocity, and particularly for studying the impingement attack experienced by condenser tubes, involves a submerged jet through which salt water or a mixture of salt water and air bubbles is made to impinge on a test specimen held in front of the jet. The latest modification of the original apparatus developed by Bengough and May (74) and described more recently by May and Stacpoole (75) is illustrated in Fig. 64 as set up at the Kure Beach (Harbor Island) Marine Test Station. The appearance of a typical group of specimens after such a test is shown in Fig. 65. LaQue and Stewart (72) have used a somewhat similar aspirator type jet test apparatus in which the air bubbles are sucked into the jet through a venturi type nozzle instead of being introduced under pressure as in the apparatus designed by May and his associates.

Along the same lines is an apparatus employed by Brownsdon and Bannister (76) in which a stream of air at high velocity is directed against the surface of a submerged test specimen.

These several types of jet test apparatus yield generally consistent results and have served very well in rating different alloys as to their abilities to resist the erosion or impingement attack encountered at the inlet ends of condenser tubes or around solids that may cause partial obstructions and peculiar turbulent effects along the lengths of tubes. The results have also been applicable to erosion problems resulting from severe turbulence around valves, fittings, connections, and sharp bends in salt water pipe lines.

Effects of velocity sometimes are aggravated by the presence of abrasive solids in suspension which complicate the carrying out of tests designed to choose materials for pump impellers and pipe lines to handle corrosive liquids at high velocities. Special apparatus for studying velocity effects in such cases has been designed and described by Fontana (**77**).

Before leaving this discussion of velocity effects, it is well to point out that while, as indicated, the study of effects of high velocity is not easy, the achievement of zero velocity in a test setup is at least as difficult.

Many investigators have made the error of assuming that by not making any attempt to move either the specimens or the testing liquid they have thereby provided zero velocity. This neglects such effects as convection currents and the streaming effects of corrosion products moving under the influence of gravity. No attempt will be made here to describe the elaborate devices that may be required to approach zero velocity in a test setup. It will suffice to point out that the most common difficulty arising from the points under discussion is that when tests are made in presumably quiet or stagnant solutions the effects of convection currents, etc., introduce an uncontrolled variable factor that makes it extremely difficult to secure reproducible results from test to test. Therefore, even when there is no practical interest in the effects of any appreciable velocity, it is desirable to provide for some controlled movement of either the specimens or the solution at some such velocity as 15 ft per min.

Up to here, attention has been directed to effects of the incidental conditions of exposure. It is equally important to be concerned with the testing solution itself. Results may be affected considerably by such features as:

1. Impurities in the chemicals used to make up the test solutions which may act to accelerate or retard corrosion and to affect one class of materials in one way and another in the opposite way, for example, ferric sulfate in sulfuric acid which would accelerate corrosion of copper and retard corrosion of stainless steel.
2. Accumulation of corrosion products which may either accelerate or retard further corrosion—this is especially important if specimens of different metals are exposed in the same vessel. As a general rule, this practice should be avoided.
3. Exhaustion of chemicals present in minute concentrations and which may have a controlling effect.

Whatever is done to take care of the points just mentioned will be determined generally by common sense based on such exploration of the possible difficulties from these sources as may be undertaken prior to setting up the details of the test procedure to be followed.

ALTERNATE IMMERSION TESTS

Some investigators (**78**) prefer alternate immersion tests over continuous immersion tests. This has led to the setting up of recommended procedures for such tests as covered by Tentative Method B 192 (**79**). Most of the comments on the total immersion test apply as well to alternate immersion tests. In addition, attention is directed to the following features that require special consideration in an alternate immersion test:

1. To assure reproducibility, from time

to time it is necessary to control the humidity of the room or chamber in which the test is carried out, since this will control the rate of drying during the time the specimens are out of the solution.

2. Materials will vary considerably amongst themselves with respect to the ratio and extent to which they will become dry while out of the liquid—in line with the hygroscopic properties of their corrosion products and the humidity of the atmosphere above the liquid.

3. High velocities cannot be achieved readily in the usual alternate immersion test setup. What might be called an alternate immersion test at high velocity can be carried out by mounting specimens on a horizontal shaft or vertical disk so that they pass in and out of the solution during such rotation. A device of this sort was described by Freeman and Tracy (**80**).

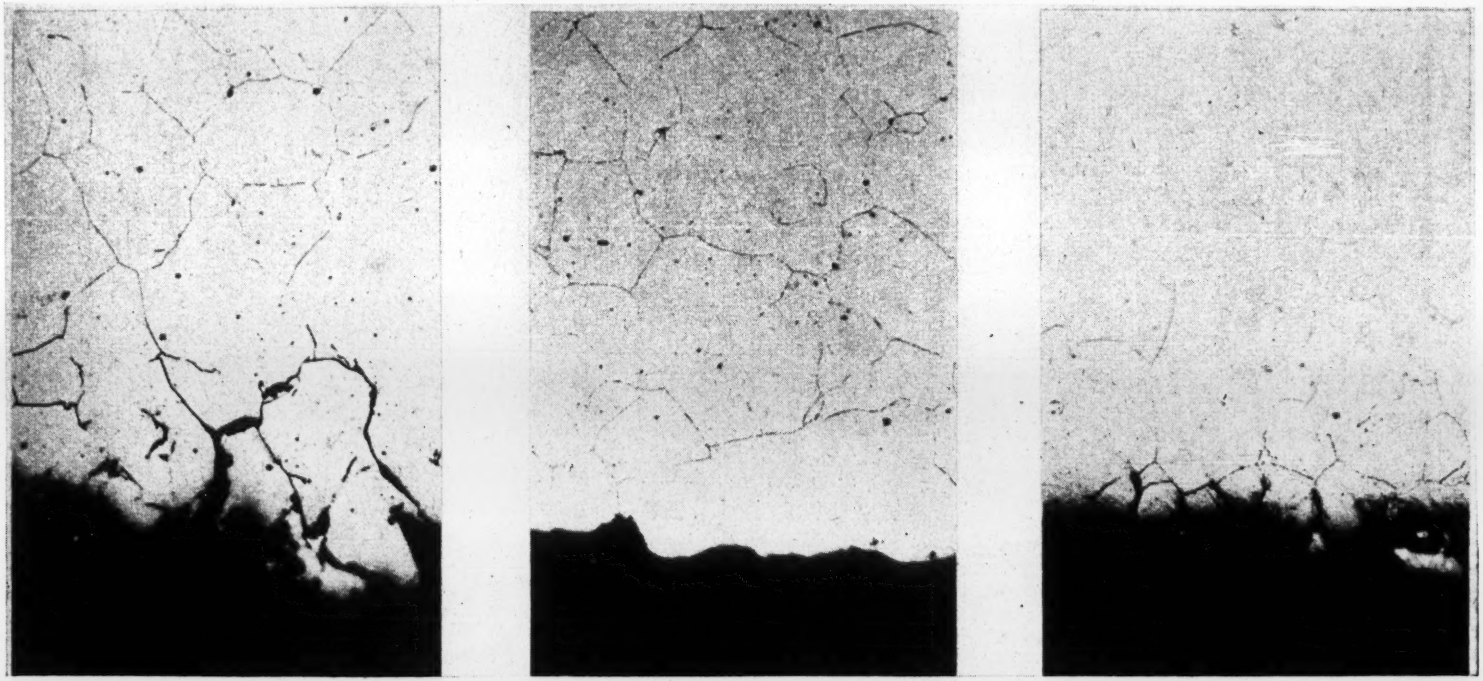
BOILING NITRIC ACID TEST

This is a special purpose test which has been very useful for its special purpose, but which, like any test that is convenient to undertake and can be made fairly quickly, has been misapplied on occasion and has thereby become subject to some criticism. Upon analysis, it will be found that the criticism is not directed so much towards the details of the test as towards its misapplication and the misinterpretation of its results.

The test was developed by W. R. Huey (**81**) and subsequently standardized (**82**) by Committee A-10. There have been a few modifications of the test procedure from time to time. Probably the most important improvement has been in the design of the apparatus which permits a large number of specimens to be tested at once (**83**), thus greatly reducing the amount of detailed laboratory attention in the carrying out of the large number of tests that are conducted for control and inspection purposes.

At the Annual Meeting in 1949, considerable attention was given the details of this test and the significance of the results in a Symposium on Evaluation Tests for Stainless Steels (**84**). At the same time, there was consideration of the acid copper sulfate test (**85**) and the nitric-hydrofluoric test (**86**). Consequently, there is no need to go into these matters in great detail here. The general situation, particularly with respect to the nitric acid test, was summed up very well in the Symposium paper by Brown, DeLong, and Myers (**87**) from which the following paragraphs are quoted:

> "It is important to emphasize that the primary purpose of the boiling 65 per cent nitric acid test is to detect the susceptibility of stainless steels to intergranular corrosion. In this sense, and only in this sense, the results are applicable to any media capable of attacking susceptible material intergranularly. The purpose of the test in the routine evaluation of stainless steel is to establish its quality, particularly with reference to its heat treatment or stabilization, so that any necessary corrective measures can be taken before it is installed as equipment. The suitability of the material to withstand general corrosion in any media other than the testing solution must be independently established by previous experience or by corrosion tests in the particular corrosives involved. The same statements apply to other tests designed to detect susceptibility to intergranular attack.
>
> "Probably the weakest point in our present knowledge of intergranular corrosion and our use of evaluation methods is lack of information on exactly which of the many and widely varied service environments for which stainless steels are employed will attack susceptible material intergranularly. Such corrosion may take place very slowly with little loss in weight or visual indication of damage until failure suddenly occurs, perhaps after several years of service. It is known that some environ-



After Exposure in Boiling Nitric Acid

After Exposure in Plant Test

After Exposure in Acid Copper Sulfate

Fig. 66.—Comparison of Intergranular Attack of 18-8 Stainless Steel in Heat-Affected Zone of Welded Test Pieces in Laboratory Evaluation Tests as Compared with No Intergranular Attack in More Corrosive Plant Test.

ments will cause rapid failure, and there is evidence that some very mild conditions will not attack even highly susceptible material in any reasonable period of time. However, a large proportion of stainless applications fall into a borderline class in which it is uncertain whether or not slow intergranular penetration will occur. Under such conditions the conservative user specifies quench-annealed or stabilized material, sometimes needlessly; the less conservative user 'takes a chance,' sometimes successfully, and sometimes with unfortunate results. In both cases more adequate knowledge would have avoided increased cost. It is probable that such information can be obtained only through an extensive field testing program involving careful observation over a period of several years of many sets of specimens chosen to be most indicative and exposed in a wide variety of service environments."

The principal points that seem to require further emphasis are:

1. Rates of corrosion observed in the boiling nitric acid provide no basis for estimating rates of corrosion in other environments nor for comparing the relative merits of different alloys in resisting attack by other media and under different conditions of exposure.
2. Attack in the nitric acid test may not be intergranular, and certain elements, such as titanium and molybdenum, may be associated with relatively high rates of weight loss in the nitric acid without the attack being intergranular. In the case of titanium in particular, the nitric acid test may not measure its ability to protect against intergranular attack, especially in other media.
3. Susceptibility to intergranular corrosion disclosed by the boiling nitric acid test will not necessarily lead to such attack in other corrosive environments.

With further reference to item 3, it is probably not surprising that intergranular attack indicated by the nitric acid test may not be encountered in environments that are much *less* corrosive than the boiling nitric acid. However, it may be surprising and therefore should be emphasized that intergranular attack may not occur in corrosive media considerably *more* aggressive than the boiling nitric acid in which susceptibility may be indicated.

This point is illustrated by the behavior of some welded specimens of 18-8 stainless steel (composition of plate, 19.17 per cent chromium, 9.48 per cent nickel, 0.08 per cent carbon). Specimens in the as-welded condition were exposed in a chemical plant for 45 days to attack by impure methyl acetate which contained about 2 per cent free acetic acid plus other organic compounds in a fractionation column which operated at about 220 F. Duplicate as-welded specimens were exposed in the laboratory to the standard boiling nitric acid test. In this nitric acid test, the weight loss was equivalent to penetration at a rate of 0.002 in. per month. In the plant test, the rate of corrosion of the welded specimens was nine times as great, that is, 0.018 in. per month. In the nitric acid test and in the acid copper sulfate test (85) the specimens suffered some intergranular attack in the heat affected zone alongside the weld as shown by the photomicrographs in Fig. 66 whereas in the mere corrosive plant test there was no evidence of intergranular corrosion in the heat-affected zone as illustrated by the photomicrograph in Fig. 66.

These results illustrate the fact that susceptibility of stainless steels to intergranular corrosion disclosed by the commonly used laboratory tests may or may not lead to this sort of corrosion in some different service environment which may be either *more* or *less* corrosive than the laboratory testing media. The importance of the corrosive environment in the occurrence of weld decay was illustrated also by the behavior of the welded stainless steel specimens in quiet and flowing sea water described elsewhere in this lecture (see Fig. 38).

On the other hand, the author is not aware of any cases where alloys shown to be free from susceptibility to intergranular corrosion (weld decay) in the standard laboratory tests subsequently suffered this sort of attack in practical service. Thus, the test errs on the conservative, or safe side, and when it has been wrong, it has tended to lead to the use of alloys too good for the job rather than not good enough—at least so far as the avoidance of weld decay difficulties has been concerned.

With increasing emphasis being placed on the need to conserve strategic materials employed to avoid weld decay difficulties and to broaden the application of the more common varieties of stainless steel, the desirability of securing more information on behavior in the evaluation tests as compared with behavior in other media has become even more important.

In this connection, the large scale program of plant tests of welded specimens in which the ASTM is cooperating with the Welding Research Council will yield very valuable results.

The author is glad that this program is to include the exposure of artificially sensitized specimens as a supplement to the specimens sensitized simply by welding. In his opinion, the making of a weld on a test specimen is a very awkward way to create the effect of a particular heat

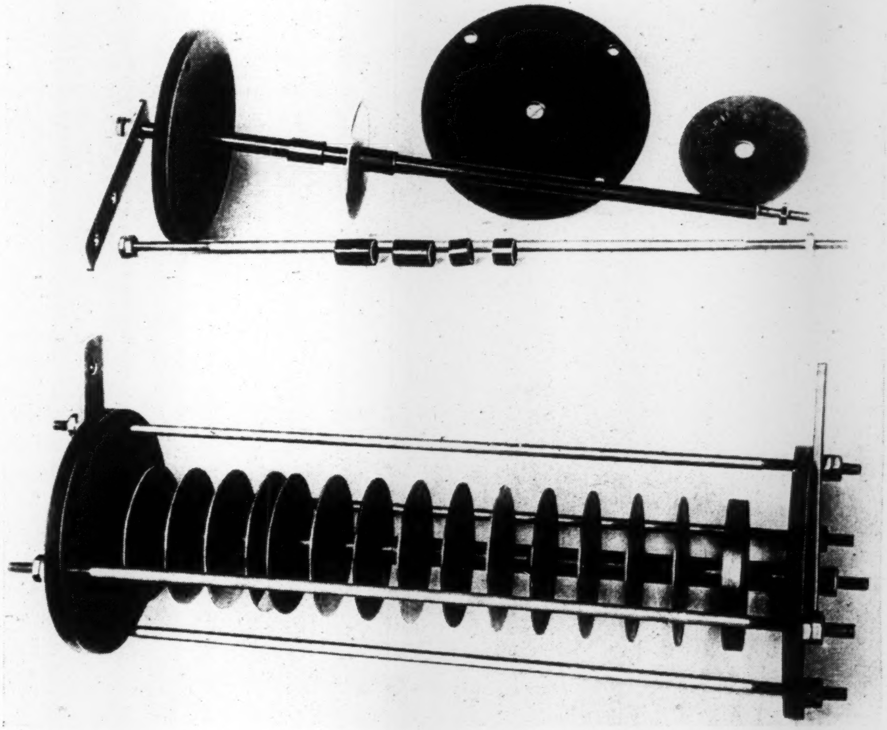


Fig. 67.—Parts and Assembly of Spool Type Specimen Holder as Used for Plant Corrosion Tests.

treatment. Such welded specimens reflect in varying degrees the peculiarities of the welder who makes the test specimens and cannot include the effects of geometry and mass on the heating and cooling associated with the welding of some particular piece of equipment. Thus, while a particular welded coupon may perform satisfactorily, there is no real assurance that all welds of the same material exposed to the same environment will be equally satisfactory. However, if an artificially sensitized specimen also behaves satisfactorily in the same test, then one can proceed with greater assurance in not taking further precautions to avoid weld decay difficulties in the use of the steel in the service towards which the test program is directed.

PLANT CORROSION TESTS

It is obviously difficult to reproduce in a laboratory all the complications of corrosive media compositions and the incidental factors that affect corrosion in process equipment. This has led to the practice of securing information on corrosion by exposing test specimens under the actual conditions of use in operating equipment. The essential features of how such tests should be undertaken are covered by Recommended Practice A 224 (88). A typical test assembly is shown in Fig. 67. This provides for electrical insulation of test specimens to eliminate galvanic effects and a degree of mechanical protection to avoid loss of specimens by reasons other than corrosion. A somewhat similar test method designed primarily for studying corrosion in pipe lines, and especially steam condensate return lines in heating systems, is covered by Method D 935 (89).

In the case of the plant tests, it is necessary also to consider the following special points in planning the tests and interpreting the results (90).

1. Specimens exposed in a vapor stream at the ambient temperature of the vapor may not encounter a corrosive condensate and will not indicate corrosion by such vapors as they condense on a metal surface.

2. Specimens exposed to hot gases that do not condense may be corroded more than surfaces used to cool such gases.

3. Specimens immersed in a liquid that is being heated may not corrode in the same way as metal surfaces used to transfer heat to the liquid. Sometimes the heated surface will corrode faster than a specimen in the hot liquid. At other times, the heated surface may corrode less through the acquisition of a protective deposit or scale that will not form on a specimen in the hot liquid.

4. Specimens exposed only in the liquid phase may not corrode the same as in the vapor phase and *vice versa*. Neither condition of exposure will show effects that may result from partial immersion or occur only at a well-defined water line or liquid level.

5. Corrosion products from adjacent specimens or derived from the equipment in which the tests are made may act either to accelerate or retard corrosion of test specimens, for example, aluminum may suffer, and stainless steels benefit from the presence of copper in such corrosion products.

6. It is extremely difficult to expose specimens so that they will encounter severe velocity effects such as may be encountered in heat exchanger tubes, pipe lines, and high-speed pumps.

Somewhat along the same line is the practice of using test specimens in the form of a component of a piece of equipment constructed of some other metal. A typical example is the installation of trial tubes in a heat exchanger. The principal sources of difficulties in such tests are the galvanic effects that may develop so as to have a serious influence on the result secured. This may be a major problem where the corrosive medium is on the outside of the tubes, but is unlikely to be serious where the corrosive liquid flows inside the tubes and any galvanic effects are unlikely to extend more than a couple of tube diameters where they can be allowed for in interpreting the results. This applies as well to tests of sections of pipe lines where, also, it is frequently practical to include insulated joints so as to eliminate galvanic disturbance of the tests.

TESTS OF PAINTS

The author has not had sufficient first-hand experience in carrying out tests of paints to permit any extensive treatment of this subject here. However, investigations in this field have been carried on by committees of the Society since its early days—starting from the large scale practical tests initiated by Committees A-5 and D-1 in 1906 and 1908.

This subject continues to receive detailed attention by the several subcommittees of Committee D-1.

In exposing painted panels to the atmosphere, the paint committees prefer to place panels at an angle of 45 deg from the horizontal instead of the 30 deg used with bare steel specimens.

The paint committees are also quite properly concerned with the study of the degradation of the paint films themselves as much as with subsequent or simultaneous deterioration of the metal being protected. To facilitate inspection of panels in an orderly and uniform manner, photographic standards have been prepared to illustrate several forms and degrees of deterioration (91). Specimens under test are rated with reference to these standards.

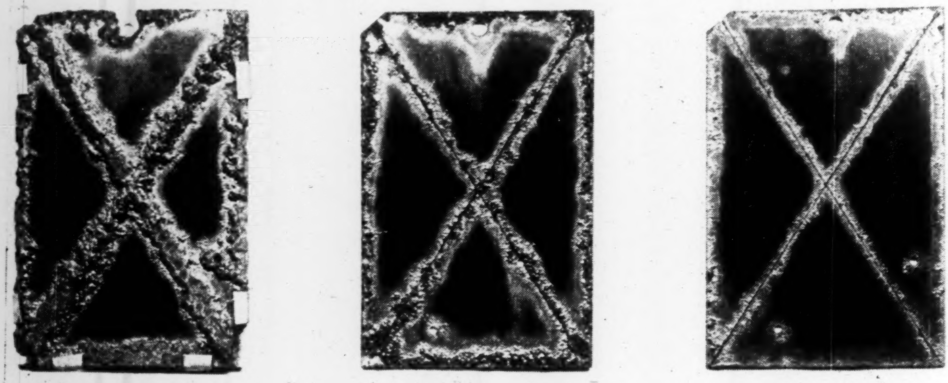
As in the study of atmospheric deterioration of metals, there is considerable interest in the use of accelerated tests for coatings, and devices for this purpose are in common use. They usually provide for exposure of the coated panels to the influence of ultraviolet light, as well as water sprays in controlled cycles designed to simulate the destructive action of sunlight and rain.

And again, as in the case of accelerated tests for metals, the reproducibility of the results from test to test and laboratory to laboratory and the correlation between these simulated weather tests for coatings and the performance of the same coatings under natural conditions of exposure are not wholly satisfactory (92, 93).

In view of the interest in what happens to the coatings, as well as what happens to the metals beneath them, the investigators of coating performance have a very difficult problem in carrying out tests in severe industrial atmospheres. Here, the panels tend to become more or less heavily coated with sooty black deposits which obscure what has happened and make inspection very difficult. One can hardly blame the investigators for seeking to find test sites that will represent the corrosive effects encountered in severe industrial areas while at the same time having an atmosphere clean enough so that the coatings to be exposed will remain clean enough to permit observations of changes in the appearances of the coatings to be observed readily throughout the progress of a test. It is the author's opinion that test sites that will provide such a combination of characteristics are rare, if not actually nonexistent. Furthermore, results of tests made under such conditions could hardly be applied to predict the performance of coatings in industrial atmospheres where the accumulation of dirt on painted surfaces is the rule.

With regard to factors in the degradation of paints, particular importance is attached to effects of natural sunlight (particularly ultraviolet rays) and effects of rain and dew. Consequently it is highly desirable if not actually necessary to provide a record of these weather factors during the progress of atmospheric tests of paints—especially if comparisons are to be made amongst test panels exposed at different places or at different times.

There has been some controversy as to the relative destructive effects of ultraviolet light and moisture. Much information on this subject was provided by the comprehensive experiments of R. Wirshing and his associates (**94**). The results were also summarized by Wirshing in a special paper presented at the Fifty-Fourth Annual Meeting of the Society under the auspices of Committee D-1.



	0.01 Cu Open-Hearth Iron	Copper Steel	Low-Alloy High-Strength Steel
Weight Loss of Unprotected Panels in 18 Months	130g	14g	7g

FIG. 68.—Effect of Composition on Corrosion Resistance of Steels and Performance of Protective Coating.

These tests showed that sunlight alone was not particularly damaging to enamels or lacquers but that a combination of sunlight and moisture in sequence caused rapid failure by chalking.

Committee D-1, through its Subcommittee XXIX, has devoted considerable attention to the preparation of steel panels to be used for testing paint coatings. The dimensions of the test specimens and the preparation of the surface have been carefully described in ASTM Tentative Method D 609 (**95**). The chemical composition of the steel to be used is not specified. Some results of recent tests by the author and his associates have indicated that under some circumstances differences in the composition of the steel or iron can have a considerable effect on the performance of paint coatings, such as on a typical automobile body finish applied to steels of different composition. Specimens were exposed in different atmospheres, but for the present purpose it will suffice to refer only to those exposed near the ocean at Kure Beach, N. C. The results secured at other test locations will be described in detail in a forthcoming paper.

The effects of composition of the steel are illustrated by the photographs of test panels in Fig. 68 which includes under the photograph of each painted panel the weight loss of unprotected specimens of the same steel or iron. It is evident that the atmospheric corrosion resistance of a steel has a great bearing on the performance of a paint system that may be applied to it. Though most carbon steel purchased at this time will contain sufficient residual copper (for example about 0.08 per cent) to give it fair resistance to atmospheric corrosion, it would appear to be desirable to exercise some control over the composition of steel to be used for paint test specimens. It also would be desirable to use the same composition of steel for panels to be used to compare different paint systems in a test program and to avoid any appreciable variations in composition—particularly with respect to copper content—from one test program to another.

Incidentally, this test also demonstrated that the superior corrosion resistance of a steel as determined by exposure of bare specimens will be realized in terms of better performance of a paint coating when used with such protection.

CONCLUSION

In this general review of corrosion testing methods that are of particular concern to members of this Society, attention has been focused on precautions that must be observed in planning and executing tests and particularly in interpreting the results. This emphasis on caution should not give the casual reader a false impression that corrosion tests are characteristically unreliable. Corrosion testing procedures used or sponsored by ASTM committees compare favorably with other kinds of tests when they are executed carefully and interpreted intelligently. That this is true is shown by the small number of cases of unexpected failures by corrosion or gross misapplication of corrosion-resisting materials. As this subject continues to receive attention by the several ASTM committees concerned with corrosion, further advances in corrosion testing methods and our knowledge of corrosion may reasonably be anticipated.

Acknowledgments:

There probably is no group of people more anxious to be helpful to one another than those engaged in the study and prevention of corrosion. All share their information unselfishly, as may be seen, for example, by the considerable amount of data contributed to this lecture by friends of the author. The names of some of these have been mentioned in the text—limitations of space will not permit listing of the many others whose help nevertheless is gratefully acknowledged.

However, particular acknowledgement is due certain of the author's associates who helped in special ways in the preparation of this lecture. These were: H. R. Copson in connection with tests in industrial atmospheres; C. Cheng with acid tests; H. T. Paterson, Jr. and R. Teel with tests in sea water and sea air; E. A. Tice in statistical analysis of data; Mrs. Eleanor Adams who prepared most of the figures and K. Henderson who made the slides used in presenting the lecture.

Finally, the author is grateful to the officials of The International Nickel Co., Inc., for their unrestrained encouragement and support of the test programs that have yielded much of the data used to illustrate the topics discussed in this lecture.

APPENDIX

STATISTICAL ANALYSIS OF ATMOSPHERIC CORROSION DATA ON COPPER-BEARING STEELS IN A MARINE ATMOSPHERE

In 1941, a large number of steels were exposed to the atmosphere at the Kure Beach, N. C. test station. In this group of steels there were nine "copper-bearing" steels produced by seven different steel companies. The analyses of these steels are shown in Table XVII. A sufficient number of sheet panels, 4 by 6 in., were exposed so that duplicate specimens could be removed for weight loss determination at 6 months and 18 months, and quadruplet specimens at 42 and 90 months.

TABLE XVII.—ANALYSIS OF COPPER-BEARING STEELS.

Steel	Composition, per cent							
	Carbon	Manganese	Phosphorus	Sulfur	Silicon	Copper	Nickel	Other
No. 1	0.06	0.26	0.007	0.032	0.01	0.19	0.06	...
No. 2	0.046	0.40	0.006	0.019	0.005	0.21	...	...
No. 3	0.08	0.34	0.008	0.023	0.01	0.22	...	...
No. 4	0.06	0.32	0.010	0.023	<0.01	0.24	...	...
No. 5	0.03	0.17	0.006	0.026	0.01	0.27	...	...
No. 6	0.026	0.24	0.009	0.035	0.004	0.27	...	...
No. 7	0.05	0.31	0.008	0.018	0.01	0.27	...	...
No. 8	0.04	0.31	0.004	0.021	0.01	0.30	0.05	...
No. 9	0.08	0.42	0.010	0.023	0.01	0.30	...	...
Range	0.03 0.08	0.17 0.42	0.004 0.010	0.018 0.035	0.005 0.010	0.19 0.30	0 0.06	...
Average	0.052	0.31	0.008	0.024	0.009	0.24	....	...

The data collected for these steels provided an opportunity for a statistical analysis to determine whether any of the steels were significantly different in performance from the others. Such an analysis was made for both the 42-month and 90-month periods. Employing the methods described in the ASTM Manual on Presentation of Data, the control chart in Fig. 17 was prepared. The limits shown were calculated from averages and standard deviations for samples of four, to discover whether the observed averages of weight loss varied among themselves by an amount greater than should be attributed to chance. A summary of averages and standard deviations is shown in Table XVIII and a typical calculation in Table XIX.

Observations:

1. From the data for 42 months exposure, steels No. 2 and No. 9 appear to be

TABLE XVIII.—WEIGHT LOSSES OF COPPER-BEARING STEELS.

Steel	Copper, per cent	Weight Loss, g per 48 sq in.			
		42 Months		90 Months	
		Average, $\overline{X}$	Standard Deviation, σ	Average, $\overline{X}$	Standard Deviation, σ
No. 1	0.19	26.25	0.46	45.27	1.32
No. 2	0.21	27.67	0.62	44.07	1.58
No. 3	0.22	25.92	0.55	43.60	0.62
No. 4	0.24	25.57	0.77	43.27	1.53
No. 5	0.27	25.95	0.62	43.52	1.58
No. 6	0.27	24.77	0.71	42.77	1.62
No. 7	0.27	24.70	0.14	42.22	1.16
No. 8	0.30	25.02	0.63	42.10	1.40
No. 9	0.30	24.30	0.26	41.35	1.18
Average	0.24				
Grand Average $\overline{\overline{X}}$		25.57		43.13	
Grand Average, $\bar{\sigma}$			0.53		1.33

Limits for samples of 4

$$\overline{\overline{X}} \pm 1.88\,\sigma$$

42 Months limits = 26.57 and 24.57.
90 Months limits = 45.63 and 40.63.

TABLE XIX.—TYPICAL CALCULATION FOR $\overline{X}$ AND σ.

Open-Hearth Steel, 0.21 per cent copper 42 Month Data	Weight Loss	
	X	X^2
No. 1	27.6	761.76
No. 2	27.1	734.41
No. 3	28.4	806.56
No. 4	27.6	761.76
	$\Sigma X = 110.7$	$\Sigma X^2 = 3064.49$
Average, $\overline{X}$	27.67	

$$\text{Standard deviation, } \sigma = \sqrt{\frac{\Sigma X^2}{4} - \overline{X}^2}$$

$$\sigma = \sqrt{766.12 - 765.73}$$

$$\sigma = \sqrt{0.39}$$

$$\sigma = 0.62$$

significantly different from the other steels, No. 2 being less corrosion resistant, and No. 9, more corrosion resistant.

2. From the data for 90 months exposure, all of the steels appear to corrode to the same extent, within limits attributable to chance. Within the range of copper content involved there does appear to be a trend towards slightly better corrosion resistance as the copper content is increased, but since none of the averages falls outside the limits there is no justification for assigning a cause for the trend.

3. The limits observed in this study are remarkably narrow, being of the order of less that ±6 per cent of the average.

References

(1) R. J. Anderson and G. M. Enos, "Coal Mining Investigations," *Bulletin No. 6*, Carnegie Inst. Technology, Pittsburgh (1923). Report of Committee B-3, *Proceedings*, Am. Soc. Testing Mats., Vol. 24, Part I, p. 281 (1924); Vol. 27, p. 281 (1927).

(2) F. Todt, "Corrosion and Residual Current. Part I—The Importance of Residual Currents in Metal Solution," *Zeitsch, Electrochem.*, Vol. 34, pp. 586–591 (1928).

(3) Sven Brennert, "Method for Testing the Resistance of Stainless Steels to Local Corrosive Attack," *Journal*, Iron and Steel Inst., Vol. 135, No. 1, p. 101P–111P (1937).

(4) Sven Brennert, "Influence of Temperature on the Occurrence of Pitting in Stainless Steel," Internat. Assn. Testing Mats., London Congress, p. 44 (1937).

(5) W. G. Renshaw and J. A. Ferree, Jr., "Passivating Characteristics of Stainless Steel," *Corrosion*, Vol. 7, No. 10, October, 1951, pp. 353–360.

(6) G. D. Bengough and R. May, "Notes on the Corrosion and Protection of Condenser Tubes," *Journal*, Inst. Metals, Vol. 32, pp. 81–256 (1924).

(7) Report of Committee U on The Corrosion of Iron and Steel, *Proceedings*, Am. Soc. Testing Mats., Vol. VII, p. 209 (1907).

(8) H. R. Copson, "A Theory of the Mechanism of Rusting of Low Alloy Steels in the Atmosphere," *Proceedings*, Am. Soc. Testing Mats., Vol. 45, p. 554 (1945).

(9) H. M. Howe and Bradley Stoughton, "The Relative Corrosion of Steel and Wrought Iron Tubing," *Proceedings*, Am. Soc. Testing Mats., Vol. VIII, p. 247 (1908).

(10) Report of Subcommittee V, Committee A-5, *Proceedings*, Am. Soc. Testing Mats., Vol. 31, Part I, p. 176 (1931).

(11) T. E. Lloyd, "'AC' A New Corrosion Test," *Journal of Metals*, Vol. 188, No. 8, September, 1950, pp. 1092–1093.

(12) T. Swinden and W. W. Stevenson, "An Accelerated Spray Test for the Determination of the Relative Atmospheric Corrodibility of Ferrous Materials," *Journal*, Iron and Steel Inst., Vol. 142, No. 2, p. 165P (1940).

(13) U. R. Evans and S. C. Britton, "Preliminary Investigation of Laboratory Corrosion Tests," First Report of Corrosion Committee, *Journal*, Iron and Steel Inst., Part I, p. 139 (1939).

(14) H. Pray and J. L. Gregg, "An Accelerated Atmospheric Corrosion Test," *Proceedings*, Am. Soc. Testing Mats., Vol. 41, p. 758 (1941).

(15) Report of Subcommittee III, Committee A-5, *Proceedings*, Am. Soc. Testing Mats., Vol. XIX, Part I, p. 181 (1919).

(16) H. S. Rawdon, "Atmospheric Corrosion Testing," Symposium on Corrosion Testing Procedures, Am. Soc. Testing Mats., p. 36 (1937). (Symposium issued as separate publication, *STP No. 32*.)

(17) C. P. Larrabee, "Effect of Specimen Position on Atmospheric Corrosion Testing of Steel," *Transactions*, Electrochemical Soc., Vol. 85, p. 297 (1944).

(18) Second Report of Corrosion Committee, *Special Report No. 5*, Iron and Steel Inst., p. 17 (1934).

(19) E. S. Taylerson, "Atmospheric Exposure Tests on Copper-Bearing and Other Irons and Steels in the United States," *Journal*, Iron and Steel Inst., Vol. 143, No. 1, p. 287 (1941).

(20) Eva Palmaer, "Apercu Succinct des Experiences Atmospherique de Longue Duree de La Commission Suedoise de Corrosion," *Metaux et Corrosion*, Vol. 23, p. 285 (1948).

(21) A. H. Woodcock, "Atmospheric Sea Salts and Corrosion," *Proceedings*, Am. Soc. Testing Mats., Vol. 50, p. 1151 (1950).

(22) N. B. Pilling and W. A. Wesley, "Atmospheric Durability of Steels Containing

Nickel and Copper," *Proceedings*, Am. Soc. Testing Mats., Vol. 40, p. 643 (1940).

(23) H. R. Copson, "Factors of Importance in the Atmospheric Corrosion Testing of Low Alloy Steels," *Proceedings*, Am. Soc. Testing Mats., Vol. 48, p. 591 (1948).

(24) B. J. Kelly, "Corrosion of Railroad Hopper Car Body Sheets," *Corrosion*, Vol. 7, No. 6, June, 1951, p. 196.

(25) V. V. Kendall and E. S. Taylerson, "A Critical Study of the ASTM Corrosion Data on Uncoated Commercial Iron and Steel Sheets," *Proceedings*, Am. Soc. Testing Mats., Vol. 29, Part II, p. 204 (1929).

(26) Report of Committee TP-1, "The Field Testing of 32 Alloys in the Flow Streams of Seven Condensate Wells," *Publication No. 50-3*, Nat. Assn. Corrosion Engrs., July, 1950.

(27) Report of Subcommittee XIV on Inspection of Black and Galvanized Sheets, *Proceedings*, Am. Soc. Testing Mats., Vol. 50, pp. 104–109 (1950). Report of Subcommittee XVI on Field Tests and Inspections of Bare and Metallic Coated Hardware, Structural Shapes, Plates, Bars, and Their Products, *Proceedings*, Am. Soc. Testing Mats., Vol. 50, p. 114 (1950).

(28) H. J. French and F. L. LaQue, "Alloy Constructional Steels," Am. Soc. Metals (1942).

(29) J. C. Hudson, "Present Position of the Corrosion Committee's Field Tests on Atmospheric Corrosion (Unpainted Specimens)," *Journal*, British Iron and Steel Inst., Vol. 148, pp. 161–215 (1943).

(30) C. P. Larrabee, "Effect of Composition and Environment on Corrosion of Iron and Steel," in special publication *Corrosion of Metals*, Am. Soc. Metals (1946).

(31) D. M. Buck, "Copper in Steel—The Influence on Corrosion," *The Journal of Industrial and Engineering Chemistry*, Vol. 5, No. 6, pp. 447–452 (1913); D. M. Buck and J. A. Handy, "Research on the Corrosion Resistance of Steel," *Ibid.*, Vol. 8, No. 3, pp. 209–216 (1916); D. M. Buck, "The Influence of Very Low Percentages of Copper in Retarding the Corrosion of Steel," *Proceedings*, Am. Soc. Testing Mats., Vol. XIX, Part II, pp. 224–237 (1919).

(32) R. B. Mears, C. J. Walton and G. G. Eldridge, "An Alternate Immersion Test for Aluminum-Copper Alloys," *Proceedings*, Am. Soc. Testing Mats., Vol. 44, pp. 639 (1944).

(33) R. F. Passano, "Controlled Data From an Immersion Test," *Proceedings*, Am. Soc. Testing Mats., Vol. 32, Part II, pp. 468-474; Discussion, pp. 475-476 (1932).

(34) Report of Committee B-3, *Proceedings*, Am. Soc. Testing Mats., Vol. 44, pp. 224 (1944).

(35) W. H. J. Vernon, "First Report to the Atmospheric Corrosion Research Committee (British Non-Ferrous Metals Research Association)," *Transactions*, Faraday Soc., Vol. 19, p. 886 (1924).

(36) G. N. Schramm and E. S. Taylerson, "The Influence of Rainfall and Smoke on the Corrosion of Iron and Steel," Symposium on Outdoor Weathering of Metals and Metallic Coatings, p. 51, Washington Regional Meeting, Am. Soc. Testing Mats. (1934). (Symposium issued as separate publication, *STP. No. 17*.)

(37) J. Dearden, "Climatic Effects on the Corrosion of Steel," *Journal*, Iron and Steel Inst., Vol. 159, July, 1948, p. 241.

(38) O. B. Ellis, "Effect of Weather on the Initial Corrosion Rate of Sheet Zinc," *Proceedings*, Am. Soc. Testing Mats., Vol. 49, p. 152 (1949).

(39) Report of Subcommittee XIV, Committee A-5, *Proceedings*, Am. Soc. Testing Mats., Vol. 50, p. 104 (1950).

(40) G. W. Quick, "Atmospheric Exposure Tests on Non-Ferrous Screen Wire Cloth," *Journal of Research*, Nat. Bureau of Standards, Vol. 14, p. 775 (1935); *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part I, p. 458 (1935).

(41) W. A. Wesley and H. R. Copson, "Weathering Behavior of Corrosion-Resistant Steel Insect Screens," *Proceedings*, Am. Soc. Testing Mats., Vol. 46, p. 652 (1946).

(42) F. L. LaQue, "Sea-Water Corrosion Tests," *Corrosion Handbook*, John Wiley & Sons Inc., New York, N. Y. p. 1060 (1948).

(43) J. C. Hudson, "Corrosion of Bare Iron and Steel in Sea Water," *Journal*, Iron and Steel Inst., Vol. 166, Part 2, p. 123 (1950).

(44) Report of Subcommittee V on Total Immersion Tests, *Proceedings*, Am. Soc. Testing Mats., Vol. 40, p. 98 (1940).

(45) H. A. Humble, "The Cathodic Protection of Steel Piling in Sea Water," *Corrosion*, Vol. 5, No. 9, p. 292 (1949).

(46) "Deterioration of Structures in Sea Water," Eighteenth Report of the Committee of the Institution of Civil Engineers, London (1938).

(47) F. L. LaQue and G. L. Cox, "Some Observations of the Potentials of Metals and Alloys in Sea Water," *Proceedings*, Am. Soc. Testing Mats., Vol. 40, p. 670 (1940).

(48) W. A. Wesley, "Controlling Factors in Galvanic Corrosion," *Proceedings*, Am. Soc. Testing Mats., Vol. 40, p. 690 (1940).

(49) W. G. Whitman and R. P. Russell, "The Natural Water Corrosion of Steel in Contact with Copper," *Industrial and Engineering Chemistry*, Vol. 16, p. 276 (1924).

(50) T. P. Hoar, "The Study of Cathodic Reactions in Metallic Corrosion," *Transactions*, Electrochemical Soc., Vol. 76, p. 157 (1939).

(51) U. R. Evans, L. C. Bannister, and S. C. Britton, "The Velocity of Corrosion from the Electrochemical Standpoint," *Proceedings*, Royal Soc., London, p. 131A, p. 355 (1931).

(52) N. D. Tomashov, "Cathodic Processes in Metallic Corrosion," *Comptes rendus de l'academie des Sciences de l'U.R.S.S.* vol. 52, p. 601 (1946).

(53) N. D. Tomashov, "The Corrosion of Metals with Oxygen Depolarization," Academy U.S.S.R., Moscow (1947); *Light Metals*, Vol. 10, p. 637 (1947), Vol. 11, pp. 8, 104, 155, 388 and 503 (1948).

(54) Paul Delahay, "A Polarographic Method for the Indirect Determination of Polarization Curves for Oxygen Reduction in Various Metals," *Journal*, Electrochemical Soc., Vol. 57, No. 6, June, 1950, p. 198.

(55) Report of Subcommittee VIII of Committee B-3 on Galvanic and Electrolytic Corrosion, *Proceedings*, Am. Soc. Testing Mats., Vol. 48, p. 167 (1948).

(56) J. A. Capp, "A Rational Test for Metallic Protective Coatings," *Proceedings*, Am. Soc. Testing Mats., Vol. XIV, Part II, p. 474 (1914).

(57) C. H. Sample, "Use and Misuse of the Salt Spray Test as Applied to Electrodeposited Metallic Finishes," ASTM Bulletin, No. 123, August, 1943, p. 19.

(58) Tentative Method of Salt Spray (Fog) Testing (B 117 - 49 T), 1949 Book of ASTM Standards, Part 2, p. 786.

(59) Report of Subcommittee IV, Committee A-10, *Proceedings*, Am. Soc. Testing Mats., Vol. 33, p. 178 (1933).

(60) E. H. Dix, Jr. and J. J. Bowman, "Salt Spray Testing," Symposium on Corrosion Testing Procedures, Am. Soc. Testing Mats., p. 57 (1937). (Symposium issued as separate technical publication, *STP No. 32.*)

(61) T. P. May and A. L. Alexander, "Spray Testing with Natural and Synthetic Sea Water. Part I.—Corrosion Characteristics in the Testing of Metals," *Proceedings*, Am. Soc. Testing Mats., Vol. 50, p. 1131 (1950).

(62) V. M. Darsey and W. R. Cavanagh, "Apparatus and Factors in Salt Fog Testing," *Proceedings*, Am. Soc. Testing Mats., Vol. 48, p. 153 (1948).

(63) William Blum, Paul W. C. Strausser, and Abner Brenner, "Corrosion Protective Value of Electrodeposited Zinc and Cadmium Coatings on Steel," *Journal of Research*, Nat. Bureau of Standards, Vol. 16, No. 2, February, 1936, p. 185.

(64) Report of Subcommittee II, Committee B-8, see p. 204.

(65) C. F. Nixon, "A Modified Salt Spray Test for Chromium Plated Zinc Base Die Castings," *Monthly Review*, Am. Electroplaters' Soc., Vol. 32, p. 1105 (1945).

(66) L. R. Voigt, "Selected Bibliography on Salt Spray Testing, 1935–1946," *Corrosion*, Vol. 4, pp. 492–500 (1948); Discussion by L. M. Flaherty, *Ibid.*, Vol. 5, p. 54 (1949).

(67) A. L. Alexander and T. P. May, "Spray Testing with Natural and Synthetic Sea Water. Part II.—A Study of Organic Coatings," *Proceedings*, Am. Soc. Testing Mats., Vol. 50, p. 1144 (1950).

(68) A. Portevin and E. Herzog, "Influence de Divers Facteurs sur les Essais de Corrosion in Milieu Humide des Aciers Ordinaires et Inoxydables," Quatorzieme Congres de Chimie Industrielle, Paris, October 21–27, 1934.

(69) Tentative Method of Total Immersion Corrosion Test of Non-Ferrous Metals (B 185 - 43 T), 1949 Book of ASTM Standards, Part 2, p. 794.

(70) W. A. Wesley, "The Total Immersion Corrosion Test," *Proceedings*, Am. Soc. Testing Mats., Vol. 43, p. 649 (1943).

(71) G. L. Cox, "Effect of Temperature on the Corrosion of Zinc," *Industrial and Engineering Chemistry*, Vol. 23, p. 902 (1931).

(72) F. L. LaQue and W. C. Stewart, "Expose de Quelques Methodes Ayant pour Abjet d'Etudier le Comportement d'Alliages en Presence d'eau de mer en Movement Rapide," *Metaux et Corrosion*, Vol. XXIII, No. 274, June, 1948, p. 147.

(73) W. H. J. Vernon, "Chemical Research and Corrosion Control: Some Recent Contributions of a Corrosion Research Group," *Journal*, Soc. Chemical Industry, London, Vol. 66, 1947, p. 137; see also *Corrosion*, Vol. 4, p. 141 (1948).

(74) G. D. Bengough and R. May, "Seventh Report to the Corrosion Research Committee of the Institute of Metals," *Journal* Inst. Metals, Vol. 32, p. 81 (1924).

(75) R. May and R. W. deVere Stacpoole, "The Jet-Impingement Apparatus for the Assessment of Corrosion by Moving Sea-Water," *Journal*, Inst. Metals, Vol. 77, Part 4, p. 331 (1950).

(76) H. W. Brownsdon and L. C. Bannister, "A Modified Impingement Corrosion Apparatus," *Journal* Inst. Metals, Vol. 49, No. 2, p. 123 (1932).

(77) Mars G. Fontana, "Corrosion," *Industrial and Engineering Chemistry*, Vol. 39, No. 6, June, 1947, p. 87A.

(78) D. K. Crampton and N. W. Mitchell, "Alternate Immersion and Water Line Test," Symposium on Corrosion Testing Procedures, Am. Soc. Testing Mats., p. 74 (1937). (Symposium issued as separate publication, *STP No. 32*).

(79) Tentative Method of Alternate Immersion Corrosion Test of Non-Ferrous Metals (B 192 - 44 T), 1949 Book of ASTM Standards, Part 2, p. 804.

(80) Discussion by J. R. Freeman, Jr. and A. W. Tracy of paper on "The Principles of Corrosion Testing," Symposium on Corrosion Testing Procedures, Am. Soc. Testing Mats., p. 32 (1937). (Symposium issued as separate publication, *STP No. 32*).

(81) W. R.Huey, "Corrosion Test for Research and Inspection of Alloys," *Transactions*, Am. Soc. Steel Treating, Vol. 18, p. 1126 (1930).

(82) Tentative Recommended Practice for Boiling Nitric Acid Test for Corrosion-Resisting Steels (A 262 - 44 T), 1949 Book of ASTM Standards, Part I, p. 874.

(83) W. B. DeLong, "Testing Multiple Specimens of Stainless Steels in a Modified Boiling Nitric Acid Test Apparatus," Symposium on Evaluation Tests for Stainless Steel, p. 211; Discussion, p. 215, Am. Soc. Testing Mats. (1950). (Symposium issued as separate publication, *STP No. 93.*)

(84) Symposium on Evaluation Tests for Stainless Steels, Am. Soc. Testing Mats. (1950). (Symposium issued as separate publication, *STP No. 93.*)

(85) Standard Specification for Corrosion-Resisting Chromium and Chromium-Nickel Steel Plate, Sheet and Strip for Fusion-Welded Unfired Pressure Vessels (A 240 - 49), 1949 Book of ASTM Standards, Part 1, p. 408.

(86) F. K. Bloom and M. E. Carruthers, "Accelerated Corrosion Testing of Chromium-Nickel Stainless Steel Weldments," Symposium on Evaluation Tests for Stainless Steels, Am. Soc. Testing Mats., p. 87 (1950). (Symposium issued as separate publication, *STP No. 93.*)

(87) M. H. Brown, W. B. DeLong and W. R. Myers, "An Appraisal of Methods for Evaluating the Corrosion Resistance of Stainless Steels," *Ibid*, p. 103.

(88) Recommended Practice for Conducting Plant Corrosion Tests (A 224 - 46), 1949 Book of ASTM Standards, Part 1, p. 865.

(89) Standard Method of Corrosivity of Industrial Water (NDHA Method) (D 935 - 49), 1949 Book of ASTM Standards, Part 5, p. 1515.

(90) F. L. LaQue and B. B. Knapp, "Planning and Interpreting Corrosion Tests," *Corrosion and Material Protection*, Vol. 2, No. 1, April, 1945, pp. 17–23.

(91) Standard Method of Evaluating Degree of Resistance to Rusting Obtained with Paint on Iron or Steel Surfaces (D 610 - 43), 1949 Book of ASTM Standards, Part 4, p. 486.

(92) Report of Subcommittee VII, Committee D-1, *Proceedings*, Am. Soc. Testing Mats., Vol. 40, p. 289 (1940).

(93) Report of Subcommittee VII, Committee D-1, *Proceedings*, Am. Soc. Testing Mats., Vol. 42, p. 294 (1942).

(94) R. J. Wirshing and W. C. McMaster, "Factors Affecting the Testing of Automotive Finishes," *Paint and Varnish Production*, September, 1951, p. 13.

(95) Tentative Method of Preparation of Steel Panels for Testing Paint, Varnish, Lacquer, and Related Products (D 609 - 50 T), 1950 Supplement to Book of ASTM Standards, Part 4, p. 84.

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON STATISTICAL ASPECTS OF FATIGUE

The papers and discussions in the Symposium on Statistical Aspects of Fatigue were presented at the Seventh Session of the Fifty-fourth Annual Meeting of the American Society for Testing Materials in Atlantic City, N. J., June 19, 1951, under the sponsorship of Committee E-9 on Fatigue.

The following papers were presented:

Introduction—R. E. Peterson
Planning and Interpretation of Fatigue Tests—Alfred M. Freudenthal
On the Statistical Nature of Fatigue—Foster B. Stulen
Statistical Analysis of Fatigue Data—Robert Plunkett

The papers together with discussions are issued as ASTM Special Technical Publication No. 121 entitled, "Symposium on Statistical Aspects of Fatigue."

THE INFLUENCE OF SURFACE ROUGHNESS ON THE FATIGUE LIFE AND SCATTER OF TEST RESULTS OF TWO STEELS*

By P. G. Fluck[1]

Synopsis

This paper presents the results of tests on fatigue specimens of two different steels prepared by six different methods of surface finishing. The tests show a marked increase in fatigue life,[2] as much as tenfold, due to polishing of the lathe-formed specimens.

Groups of twelve similar specimens were tested to provide information on the scatter or dispersion of test results. The specimens produced by the special method of gentle grinding described gave high and uniform results even though rather large longitudinal scratches remained on the specimens.

In the past, many experiments have been carried out to determine the influence of various factors such as surface roughness, size of specimen, and speed of test on the fatigue limits of various materials. Work by Kommers and Moore (**1**),[3] Thomas (**2**), Horger and Niefert (**3**), Johnson (**4**), and others has established the damaging influence of surface irregularities, but their work was concerned mainly with fatigue limit values. Discussions by Thum of a paper by Kommers (**5**) and by Schapiro and Lewis, of Found's paper (**6**) clearly illustrate the importance of the fatigue life or sloping portion of the *S-N* curve in practical design with a given material. It has become apparent that more information is needed regarding the various factors which influence fatigue life to aid in the design of parts which are intended to operate for a limited life at stresses above the fatigue limit.

A treatment which has a certain effect on the fatigue limit of a given material may have an opposite effect on the fatigue life of the material and practically always will have a different percentage effect on life and limit values.

Kommers (**5**) has illustrated this idea for one factor in his statement, "While understressing plus coaxing may increase the endurance limit from 25 to 30 per cent, it is shown that such stressing may increase the endurance life at higher stresses as much as 23,000 per cent."

S-N curves for shot-blasted and polished specimens published by Lessells and Murray (**7**) show that for SAE 9260 steel the fatigue limit was slightly lower for shot-blasted than for polished specimens but the fatigue life was much greater. In discussing the Lessells and Murray paper, Clark (**8**) states, "In considering the curves shown for SAE 9260 steel in Fig. 6, we are prompted to remark in the interest of emphasis that, though the difference in endurance limit between the shot-blasted and polished specimens is of small order, the shot-blasted specimens have many times

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[2] In this paper the term "fatigue life" is used to refer to life at stresses above the fatigue limit. In general, the paper does not consider life at stresses very near the fatigue limit.

[3] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 591.

the life of the polished specimens at stress values above the endurance limit." The shot-blasted specimens of SAE 1045 steel (heat treated) had a lower fatigue limit but greater fatigue life than the polished specimens. Shot-blasted specimens of annealed SAE 1045 steel, SAE X4340 steel, and Armco Iron had values of both fatigue limit and fatigue life higher or about equal to the corresponding values for polished specimens.

S-N curves published recently by Tarasov and Grover (9) show several interesting examples of the possible difference in effect on fatigue limit and fatigue life which may result from a given treatment. Tests of flat-bar specimens of ball-bearing type steel of Rockwell hardness C 59 showed that severely ground and hand-polished specimens had a fatigue limit about 25 per cent lower than gently ground specimens, but their fatigue life at 120,000 psi maximum stress was four times as great as the gently ground specimens. In the case of round-bar tests of the same steel, hand polishing of gently ground specimens caused a reduction of fatigue limit of about 10 per cent and an increase of fatigue life at 160,000 psi maximum stress of about 50 per cent.

The examples cited illustrate the fact that the influence of a given factor on fatigue limit may be a poor indication of its influence on fatigue life. The present paper discusses the effect of surface roughness on fatigue life. Specimens of quenched-and-tempered SAE 3130 and annealed SAE 1035 steel were prepared with six different surface finishes ranging from lathe-formed to superfinished surfaces. Specimens of each type were tested at each of two stress levels above the fatigue limit. All specimens were tested with Krouse rotating cantilever-beam machines operated at 10,000 rpm.

In any study of fatigue life the problem of the scatter of test results is encountered. Specimens that are presumably alike and tested at the same unit stress show greatly different fatigue life values. In this investigation, groups of twelve similar specimens were tested under similar conditions to provide information regarding the scatter or dispersion of test results. An attempt was made to select a type of surface finish which could be produced at low cost but which would give fatigue life test results which were both high and uniform.

Steel Properties and Specimen Preparation

The SAE 1035 steel was donated by the Inland Steel Co. in the form of $\frac{5}{8}$-in. round bars 4 ft long. All bars were obtained from one billet of steel from the lower portion of an ingot and were annealed at the Inland Steel Co. plant. The bars were annealed for 2 hr at 1540 F and furnace cooled to 600 F in 18 hr, then air cooled. No cold straightening was done after annealing.

TABLE I.—MECHANICAL PROPERTIES OF STEELS.

	SAE 1035	SAE 3130
Yield point, psi	39 300	136 000[a]
Tensile strength, psi	69 700	143 000
Elongation in 2 in., per cent	32	20
Reduction of area, per cent	50	66
Rockwell hardness	B 69	C 30
Approximate fatigue limit, psi (hand polished specimens)	31 500	78 000

[a] 0.001 in. per in. offset.

The SAE 3130 steel was purchased on the open market as $\frac{1}{2}$-in. round hot-rolled bars 16 ft long. The bars were cut into 24-in. lengths and hardened by heating at 1525 F for 40 min, followed by quenching in oil at room temperature. About 6 hr after the hardening treatment the bars were tempered by heating at 1000 F for 55 min, followed by quenching in oil at room temperature.

Microscopic examinations showed that the SAE 1035 steel had a uniform structure of pearlite and ferrite and the SAE 3130 steel had a uniform tempered martensitic structure. Table I lists the mechanical properties of the two steels.

Each specimen was roughed down with roller-follower cutters in a turret lathe and the reduced central portion formed by a forming tool of 2-in. radius fed at right angles to the axis of the specimen. When used without a further finishing operation, these specimens are referred to as "lathe-formed."

The "partly hand-polished" and "hand-polished" specimens were polished by holding progressively finer grades of emery cloth against rough specimens as they were rotated in a lathe. The finest cloth used on the partly hand-polished specimens was No. 320 emery, and the finest cloth used on the hand-polished specimens was Norton crocus cloth No. 4N6. Scratches remaining on the surface of these specimens were circumferential.

The "ground" specimens for the program were prepared on a grinding and polishing machine specially arranged for the job. The specimen was rotated at 1300 rpm and was polished by an 80 grit wheel approximately 4 in. in diameter rotated at 4600 rpm. The grinding wheel spindle was above and at right angles to the specimen axis. The rotating specimen was passed back and forth beneath the grinding wheel as the wheel was fed downward onto the specimen. An ample supply of coolant was kept flowing over the grinding wheel and specimen during the grinding. The wheels wore down rather rapidly and were dressed with a diamond at frequent intervals so that loading of the wheel was not excessive. The surfaces of the ground specimens were relatively rough but the surface scratches were very nearly longitudinal.

The "ground and polished" specimens were prepared from the ground specimens by polishing with a 320 grit rubber-bonded polishing wheel in the special machine used for grinding. An ample supply of coolant was used in the polishing operation, and only a small amount of material was removed to avoid overheating of the surface. Any polishing scratches remaining on the specimen were longitudinal.

The "superfinished" specimens were prepared in the laboratories of the Gisholt Machine Co. It must be noted that although the specimens were prepared on a superfinishing machine by the superfinishing technique, the shape of the specimen does not lend itself well to this process; so the specimens probably are not the best examples of high-class superfinishing. The scratches remaining on a superfinished surface have a random orientation.

SURFACE ROUGHNESS MEASUREMENTS

A Brush surface analyzer was used to make longitudinal surface roughness measurements, and a profilometer was used for transverse measurements. The longitudinal measurement is the more important because it is influenced by circumferential stress-raising scratches, but the transverse or circumferential measurement is also reported because it helps to define the general surface characteristics.

The tracer head of the surface analyzer consists of a rounded support button and a small cone-shaped diamond with a tip rounded to a radius of 0.0005 in. Because the rounded support button has too large a contact surface to move up and down in the small surface scratches, there is motion of the tracer diamond relative to the support button as the diamond moves in and out of the scratches while the tracer head is drawn across the surface to be analyzed.

The motion of the diamond point distorts a Piezo crystal which generates a minute electric current proportional to the distortion. The current is amplified and conducted to a second Piezo crystal which distorts and moves a pen on a moving strip chart.

The instrument is provided with a meter which operates on the principle of an ac wattmeter and indicates the root mean square (rms) of the depth of surface scratches directly in microinches.

TABLE II.—FATIGUE LIFE OF SAE 1035 STEEL SPECIMENS.

Type of Finish	Surface Roughess Measurement, microinches		Median Fatigue Life, cycles	Quartile Scatter Factor, per cent
	Longitudinal	Circumferential		
Unit Stress, 38,000 psi				
Lathe-formed	65	18	258 000	24
Partly hand-polished	16	19	728 000	12
Hand-polished	6	10	790 000	24
Ground	13	95	836 000	28
Ground and polished	3	6	1 260 000	44
Superfinished	8	7	848 000	19
Unit Stress, 36,000 psi				
Lathe-formed	65	18	566 000	10
Partly hand-polished	16	19	1 555 000	19
Hand-polished	6	10	1 670 000	25
Ground	13	95	3 598 000	51
Ground and polished	3	6	3 365 000	195
Superfinished	8	7	1 822 000	16

The profilometer, on the other hand, does not have a plotting device, so the only reading obtained is from the meter. The profilometer tracer head has two support buttons rather than one as in the Brush surface analyzer. In the profilometer the current is generated in a tiny coil attached to the shaft of the diamond tracer. The coil moves in the field of a permanent magnet as the tracer point moves up and down.

It was not possible to use the driving mechanism of the Brush surface analyzer to drive the tracer head for making transverse measurements. The centering holes in the ends of each specimen were polished and the specimen mounted on centers. The tracer head was rested on the specimen at mid-length and the specimen rotated slowly by hand. Readings obtained in this manner are not completely dependable but give a comparison of the longitudinal scratches in different types of specimens.

The longitudinal and circumferential surface roughness measurements are listed in Tables II and III. The roughness values reported are based on several check readings on three samples of each type of finish.

TABLE III.—FATIGUE LIFE OF SAE 3130 STEEL SPECIMENS.

Type of Finish	Surface Roughness Measurement, microinches		Median Fatigue Life, cycles	Quartile Scatter Factor, per cent
	Longitudinal	Circumferential		
Unit Stress, 95,000 psi				
Lathe-formed	105	15	24 000	43
Partly hand-polished	6	4	91 000	20
Hand-polished	5	3	137 000	27
Ground	7	45	217 000	19
Ground and polished	2	6	234 000	47
Superfinished	7	8	212 000	46
Unit Stress, 88,000 psi				
Lathe-formed	105	15	54 000	24
Partly hand-polished	6	4	280 000	27
Hand-polished	5	3	311 000	61
Ground	7	45	368 000	24
Ground and polished	2	6	690 000	77
Superfinished	..	..	..	..

FATIGUE LIFE TESTS

Tables II and III list the median fatigue life values for each group of specimens tested. The median values were used because they are not unduly influenced by a few unusually high or low test results. Four of the 276 specimens tested withstood 20,000,000 cycles without failure and were assumed to have infinite fatigue life values; hence the determination of mean values would be uncertain.

An examination of Tables II and III shows that for both the SAE 1035 steel and the SAE 3130 steel the fatigue life was increased as the surface of the specimen was made smoother. The median fatigue life of the SAE 3130 steel specimens was increased about tenfold at both 95,000 psi and 88,000 psi by grinding and polishing the lathe-formed specimens. The SAE 1035 steel specimens showed a fivefold increase at both 38,000 psi and 36,000 psi. The specimens with intermediate types of finish exhibited a smaller increase in fatigue life than did the ground-and-polished specimens, but the effect of reducing surface roughness was very marked. The reduction of fatigue limit due to circumferential surface scratches has been well established by many investigators but the percentage difference is relatively small.[4]

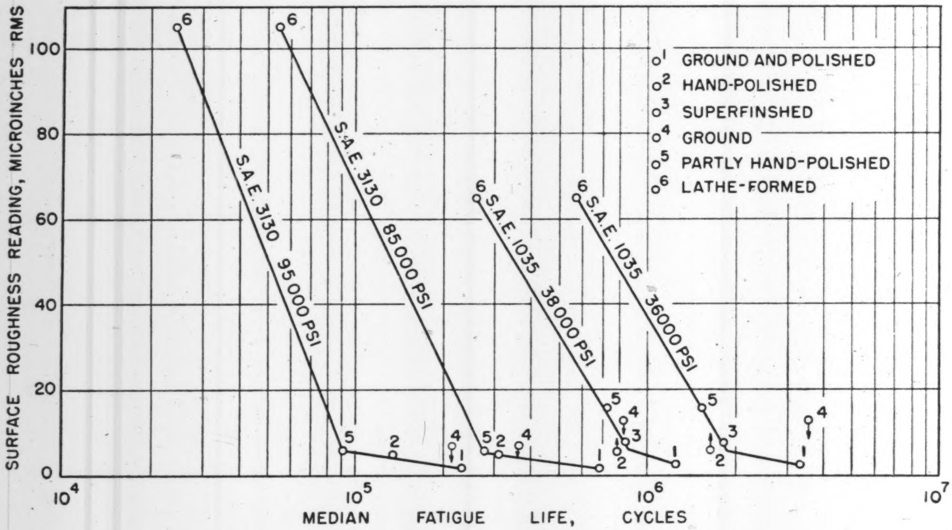

Fig. 1.—Effect of Surface Roughness on Median Fatigue Life.

The curves of Fig. 1 were plotted to illustrate graphically the increase of fatigue life with the removal of circumferential surface scratches. The surface roughness readings plotted are the longitudinal readings taken with the Brush surface analyzer and are a measure of circumferential stress-raising scratches. Each plotted point is the median value for a group of twelve specimens. The fatigue life values are plotted to a logarithmic scale, so the range of values for each curve tends to be minimized. The logarithmic scale was chosen to facilitate representation of the widely different life values on a single sheet.

The comparison of surface finish on the basis of longitudinal roughness readings only is not completely justified for specimens produced by radically different techniques, but the four plotted curves show a definite trend. It appears that the polishing of specimens to surface roughness readings below 6 microinches causes an extremely great increase in fatigue life. In the case of the SAE 3130 steel, this effect is illustrated by the hand-polished and the ground-and-polished specimens, but in the case of the SAE 1035 steel only the ground-and-polished specimens have surface roughness readings below 6 microinches.

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[4] Moore and Kommers (1) report about 20 per cent difference in the fatigue limits of rough-turned and highly polished specimens of quenched-and-drawn medium-carbon steel.

The fatigue life of the ground specimens falls above the plotted curve in each case. The author believes that the relatively deep longitudinal scratches on the ground specimens adversely affected the tracer point motion so that the Brush surface analyzer gave longitudinal surface finish readings which were too high. This effect would shift the plotted points up from their proper positions.

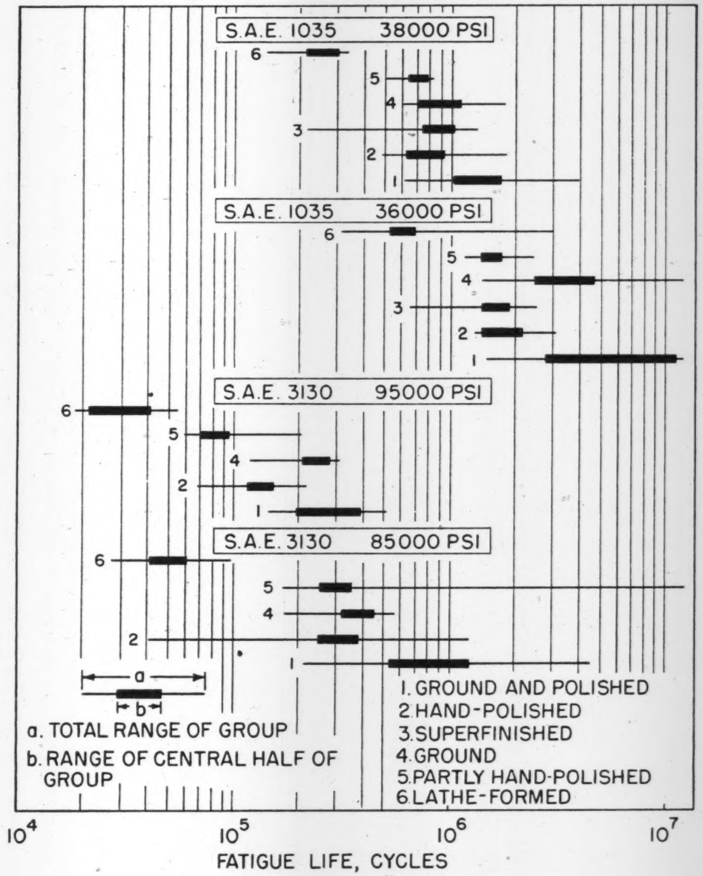

FIG. 2.—Scatter of Test Results.

The dispersion of test results for the different surface conditions and test stresses is indicated by Fig. 2 and the quartile scatter factors[5] listed in Tables II and III.

Comparison of the scatter factors for the two groups of similar specimens tested at different stresses shows that more scatter is experienced at the lower stress. This effect has been well established by other investigators. The lathe-formed specimens in this program were a marked exception to the general trend, and the superfinished specimens were a less pronounced exception.

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[5] The quartile scatter factor is one-half the range between the lower and upper quartile values expressed as a percentage of the median value. Fifty per cent of the test results of a group fall between the lower and upper quartile values. For groups of twelve specimens the quartile values are taken midway between the third and fourth highest and the third and fourth lowest test results; hence the quartile range is slightly greater than the range of the central 50 per cent of the test results.

The greater scatter of results experienced as the fatigue limit is approached makes comparison of the scatter factors for the different groups of specimens tested at the same stress misleading. Because the smooth specimens have higher fatigue limits than the rough specimens, they are being tested closer to their fatigue limits, and for that reason would be expected to show greater scatter of results than the rough specimens. A more valid comparison of scatter factors might have been obtained if the groups of specimens had been tested at some fixed percentage (for example 10 per cent) above the fatigue limit for the group, but such tests would not give comparable fatigue life values.

The ground specimens showed the least scatter of all the different surface finishes of SAE 3130 steel despite the fact that the ground specimens showed higher fatigue life values and presumably were being tested closer to their fatigue limit than most of the other types of finish. This effect is very encouraging because it indicates that specimens can be produced which will give uniform results without high-class polishing. The uniformity of results for the ground specimens of SAE 1035 steel does not compare as favorably with the other types of surface finish as in the case of the SAE 3130. The test stress of 36,000 psi appears to be close to the fatigue limit of the SAE 1035 steel in the ground condition, so great scatter would be expected. Additional tests are planned with this relatively inexpensive type of surface finishing.

The ground-and-polished specimens showed the greatest scatter of test results in each series of tests. It does not appear that this is due entirely to the fact that these specimens are closer to their fatigue limit values than specimens with other surface finishes. The greater scatter of test results may be inherent in the very smooth specimens or it may be due in part to slight nonuniformity of the polished surface.

CONCLUSIONS

1. The fatigue life of specimens of two different steels was greatly increased by reducing the size of circumferential scratches. A tenfold increase in the fatigue life of hardened and tempered SAE 3130 steel was accomplished by grinding and polishing the lathe-formed specimens. The fatigue life of the annealed SAE 1035 specimens was increased by a factor of five.

2. The polishing of specimens to surface roughness readings below 6 microinches caused an extremely great increase in fatigue life. The ground specimens showed unusually high fatigue life values, which indicates that the true longitudinal surface finish readings may have been lower than indicated by the Brush surface analyzer.

3. Longitudinal scratches, even though relatively large, did not greatly reduce the fatigue life of the two steels tested.

4. The scatter of test results was greater at low stress than at high stress for all types of specimens tested except the lathe-formed and superfinished specimens.

5. The ground specimens used in this test program were produced at relatively low cost and showed promise of giving high and uniform test values.

Acknowledgment:

The tests reported in this paper were a portion of the research carried out by the author to satisfy the requirements for the Ph.D. degree at the University of Wisconsin. The author is especially grateful to J. B. Kommers who directed the program. Financial support was provided by the University of Wisconsin Engineering Experiment Station and the Department of Mechanics.

REFERENCES

(1) J. B. Kommers and H. F. Moore, "An Investigation of the Fatigue of Metals," *Bulletin 124*, University of Illinois Engineering Experimental Station (1921).

(2) W. N. Thomas, "Effect of Scratches and of Various Workshop Finishes Upon the Fatigue Strength of Steel," *Engineering*, Vol. 116, p. 449 (1923).

(3) O. J. Horger and H. R. Neifert, "Effect of Surface Conditons on Fatigue Properties," *Preprint No. 52*, Am. Soc. Metals (1940).

(4) J. B. Johnson, "Dependence of Aviation on Metallurgy," *Metals and Alloys*, Vol. 1, p. 450 (1930).

(5) J. B. Kommers, "The Effect of Overstressing and Understressing in Fatigue," *Proceedings,* Am. Soc. Testing Mats., Vol. 43, p. 749 (1943).

(6) G. H. Found, "The Notch Sensitivity in Fatigue Loading of Some Magnesium-Base and Aluminum-Base Alloys," *Proceedings*, Am. Soc. Testing Mats., Vol. 46, p. 715 (1946).

(7) J. M. Lessells and W. M. Murray, "The Effect of Shot Blasting and Its Bearing on Fatigue," *Proceedings*, Am. Soc. Testing Mats., Vol. 41, pp. 659–673 (1941).

(8) H. H. Clark, *Ibid.*, discussion p. 675.

(9) L. P. Tarasov and H. J. Grover, "Effects of Grinding and Other Finishing Processes on the Fatigue Strength of Hardened Steel," *Proceedings*, Am. Soc. Testing Mats., Vol. 50, p. 668 (1950).

DISCUSSION

MR. J. M. LESSELLS[1].—This is an interesting paper but it is to be regretted that the author did not, in addition to studying the effect of surface roughness on the fatigue life, give us some data on the endurance limit. He does refer to the classical work of Thomas, which broadened our understanding of crack growth by reference to the endurance limit of a plain polished specimen. By use of special technique for measuring roughness, Thomas was the first to analyse the effect of surface roughness on the fatigue strength.

Another point is that no reference is made to the interesting work of the late Mr. de Forest on crack growth. He studied the effect of surface finish, both as regards crack genesis and subsequent growth, so possibly some reference to this should be made by the author.

MR. J. T. RANSOM[2] (*by letter*).—The title of Mr. Fluck's paper might more correctly be "The Influence of Surface Finishing Methods" rather than "The Influence of Surface Roughness." The various grinding and polishing procedures employed probably produced different intensities of at least three effects. These are surface roughness, surface residual stresses, and surface hardening through cold work and martensite formation. Without experimental evidence that the last two effects did not occur, we do not believe that the differences in fatigue life can be attributed entirely to the differences in surface roughness. Perhaps Mr. Fluck has additional information on these points which will justify his neglect of them.

The author is to be commended for conducting his investigation with a larger number of specimens than is customary. However, having done this, advantage has not apparently been taken of the opportunity to test the certainty or validity of his conclusions by use of common statistical methods. In particular, we believe that the conclusions with regard to the scatter produced by different finishing processes are open to question. This suspicion arises from the fact that a sample of twelve specimens does not allow a very reliable estimate of the true variability of the process. Thus, a comparison of the estimates of variability as though they represented true values may result in erroneous conclusions. The differences may be the result of sample variations.

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[1] Associate Professor of Mechanical Engineering, Massachusetts Institute of Technology, Cambridge, Mass.

[2] Materials of Construction Section, Engineering Research Lab., E. I. du Pont de Nemours and Co., Wilmington, Del.

There are available in the common texts on statistics a "variance ratio test" for comparing the scatter of two samples and a "homogeneity of variance test" for comparing the scatters of several samples. By use of these tests it can be said, for example, that the probability of the observed differences being due to sampling variations is so much. Thus, to each conclusion may be attached a significance level. In using these significance tests, it is important to realize the assumption of normality of data. All indications to date are that the logarithm of life to failure is distributed normally. Therefore all calculations must be in terms of the logarithm of life to failure.

One difficulty which will be encountered in data such as that of Mr. Fluck is the treatment of specimens which "run out." This is no doubt the reason for his use of quartile values rather than standard deviations. If a large sample is available there are ways to convert quartile values into standard deviations for a normal distribution. However, I am not aware of any method available for small samples such as used here. If these methods have been worked out, a valuable service can be rendered the society by bringing these methods to the attention of Committee E-9 on Fatigue.

MR. P. G. FLUCK (*author's closure*).—Mr. Lessells has commented on the lack of fatigue limit data in this paper. It is the author's belief that, at least by comparison, the effect of surface roughness on fatigue limit has been rather thoroughly investigated but more fatigue life tests are needed. The list of references given with the paper indicates that considerable information is available concerning the influence of surface roughness on fatigue limit, but the list is by no means complete. Omission of reference to the work of de Forest and others does not imply that their work was unimportant.

Mr. Ransom has pointed out that the groups of twelve specimens used may be too small to allow reliable evaluations of scatter and that the surface finishing methods used may have influenced the specimen properties through cold work, formation of martensite, and induction of surface residual stresses. The author agrees with both of these statements but feels that, despite these limitations, certain trends are established. The usual precautions of providing ample coolant and taking light cuts during machining and grinding were observed.

Mr. Ransom has commented on the uncertainties in the usual statistical treatment of fatigue results containing specimens which "run out." The author feels that the use of the median and quartile values in the statistical treatment of fatigue life tests has merit because it avoids the extreme effect of a few very large life values and the impossibility of handling "infinite or run out" values in the determination of mean, standard deviation, and related values. The statistical treatment in this paper is admittedly crude and less common than the method based on mean values but it appeared to be a first step toward a practical method of handling the very special statistical problem of fatigue life tests.

EFFECT OF RESIDUAL STRESS ON THE FATIGUE STRENGTH OF NOTCHED SPECIMENS*[1]

By D. Rosenthal[2] and G. Sines[2]

Synopsis

Previous investigations have established that the residual stress definitely affects the fatigue strength of metal parts, but no quantitative relations have been derived to predict the actual performance. In the present work an attempt was made to set up a basis for such a prediction by overcoming two of the major obstacles: (1) the presence of other factors, such as cold working or heat treatment, which are generally involved in the process of inducing residual stress and (2) a partial relief of residual stress which can take place during fatigue testing.

The tests were performed on notched 61S aluminum alloy in the precipitation hardened, 61S-T6 condition and in a softened condition. Residual stress, both compressive and tensile, has been set up at the base of the notch by a process of overstressing which created only negligible cold working, and the X-ray stress technique was used to follow up the change of residual stress during testing. While no change of residual stress was found in the 61S-T condition, in the softened condition the application of the fatigue load nullified any significant difference between stress-free and prestressed specimens. Accordingly, the fatigue strength of the hard alloy was influenced appreciably by the induced stress—increased in the case of residual compression and decreased in the case of residual tension—while it remained unchanged in the case of the soft alloy. A quantitative prediction of these results was made by combining X-ray data with a modified Goodman diagram of the unnotched material.

The influence of the residual stress on fatigue has been reviewed recently in some detail (1).[3] From this review it can be concluded that the residual stress definitely is a factor, but except for a few instances the proof is neither direct nor is it of a quantitative nature. That is, on the basis of the available information no quantitative prediction can be made as to the influence of the residual stress on fatigue. Two major difficulties generally exist when such prediction is attempted:

1. Along with the residual stress other changes, structural or physico-chemical, have accompanied the process of setting up the residual stress.

2. Depending on the magnitude of the applied load, the residual stress has been modified more or less during the fatigue testing.

Obviously, the above two factors must be accounted for in any theory which aims at a quantitative treatment of the subject.

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* Presented at the Fifty-fourth Annual Meeting of the Society, June 18–22, 1951.

[1] Part of the results were taken from an M.S. thesis by G. Sines, submitted to the University of California Department of Engineering in July, 1949.

[2] Department of Engineering, University of California, Los Angeles, Calif.

[3] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 608.

In the series of tests which are described and discussed in the present paper, the influence of the first factor has been minimized by an appropriate method of setting up the residual stress. The second factor has been controlled by the X-ray stress technique which by being non-destructive enables one to follow the change of the residual stress during testing.

The present work is a continuation of a study started several years ago (2) and it is far from being completed. But the results, even though fragmentary, appear of sufficient interest to be reported at this time. It will be shown that on the basis of the results obtained so far, it is possible to predict the influence of the residual stress on fatigue with a reasonable degree of accuracy.

TABLE I.—COMPOSITION AND MECHANICAL CHARACTERISTICS OF THE ALUMINUM ALLOY 61S.

Chemical Analysis (Nominal):

Copper, per cent	0.25
Chromium, per cent	0.25
Silicon, per cent	0.6
Magnesium, per cent	1.0

Mechanical Characteristics:

	61S-T[a]	61S-O*[b]
Yield point (0.2 per cent offset), psi	40 000	16 000
Tensile strength, psi	45 000	25 700
Elongation, 2-in. gage length, per cent	12	20
Fatigue strength, psi	13 500 at 5 × 10^8 cycles	11 500 at 10^7 cycles

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[a] Supplied by the manufacturer.

[b] Determined by the Engineering Department, UCLA.

As previously, the investigation has been carried out with notched rather than uniform specimens. There are two reasons for this choice:

1. The stress concentration created by the notch enables one to set up high residual stresses with a minimum amount of plastic deformation.
2. The inception of crack is confined to a point at which the induced residual stress is at a maximum.

Then also, results bearing on the notch-fatigue behavior are likely to have more practical value, since machine parts and structures usually have surface irregularities which act like notches and often are the points of initiation of fatigue cracks.

EXPERIMENTAL PROCEDURE

Material and Specimens:

For reasons which will appear later, it was desirable to use two sorts of specimens: one with a high yield point - fatigue strength ratio and the other with low yield point - fatigue strength ratio (hereafter designated as Y.P. - F.S. ratio). The material selected for this purpose was the aluminum alloy 61S whose composition is given in Table I. In the as-received condition, which is the precipitation-hardened condition 61S-T6, the alloy has a relatively high Y.P. - F.S. ratio. But by a low-temperature annealing (softening) it is possible to decrease the yield point without much changing the fatigue limit and thereby obtain a low Y.P. - F.S. ratio. The treatment consisted of maintaining the alloy at 600 F for 30 min and slow cooling in furnace, at a rate of 120 F per hr. In addition to decreasing the Y.P. - F.S. ratio the treatment was aimed at removing a small but variable amount of residual stress, up to 4000 psi, which the material contained as received.

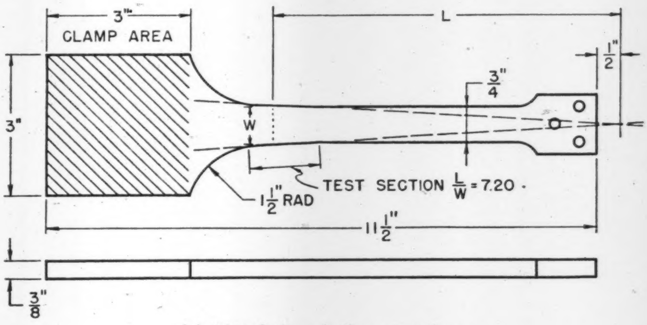
The mechanical characteristics of the 61S - T and the softened alloy, referred to hereafter as 61S - O*, are given in Table I.

Prior to the heat treatment, the specimens destined for the fatigue testing were machined to the desired shape and dimensions. They were of two types:

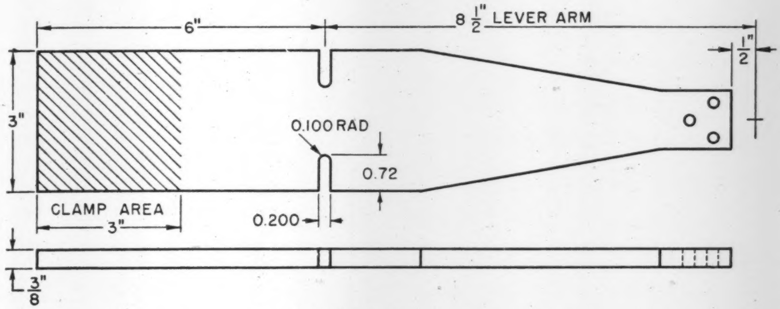
1. Notch-free specimens of the type shown in Fig. 1(*a*) for the determination of the fatigue characteristics of the material.
2. Notched specimens of the type shown in Fig. 1(*b*) for the study of the influence of the residual stress on

fatigue. The geometry of the notch and the dimensions of the notched section are very nearly the same as those used in a previous work (2, 3).

The dimensions of the specimens in the critical area were measured to within 0.001 in. Since the 61S-O* specimens were stress relieved after machining, no surface preparation was necessary for the taking of X-ray diagrams in these specimens. The as-received 61S-T specimens, however, were subjected to the proper surface preparation described elsewhere (9) aimed at the removal of stresses, induced by machining, and excessive roughness produced by etching.



(*a*) Notch-free fatigue specimen.



(*b*) Notched fatigue specimen.

FIG. 1.—Fatigue Specimens.

Inducing Residual Stress:

The residual stress was set up by subjecting the notched specimens to loads[4] of sufficient magnitude to deform plastically (permanently) the metal around the notch. Because of stress concentration, the fibers near the notch exceed the yield point sooner than the rest of the notched section. Upon the release of the load these fibers remain elongated in the case of tensile loads and shortened in the case of compressive loads. Therefore, they are acted on by the surrounding material which causes residual compressive stresses in the first case and tensile stresses in the second case. The amount and distribution of the residual stress can be controlled by varying the load. It has been shown (3) that by this procedure residual stress almost as high as the yield point of the material can be set up without deforming the metal more than a fraction of 1 per cent. Thus, the effect of cold working can be disregarded. The magnitude of the load, either tensile or compressive used for this purpose, corresponded to an average stress on the notched section of about 0.8 yield point. To minimize the effects of eccentric loading, the specimens were pulled by means of special links. Buckling was prevented by compressing the specimens between steel plates with an interposition of rubber pads to minimize friction and lateral pressure.

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[4] The loads were tensile for residual compressive stress and compressive for residual tensile stress.

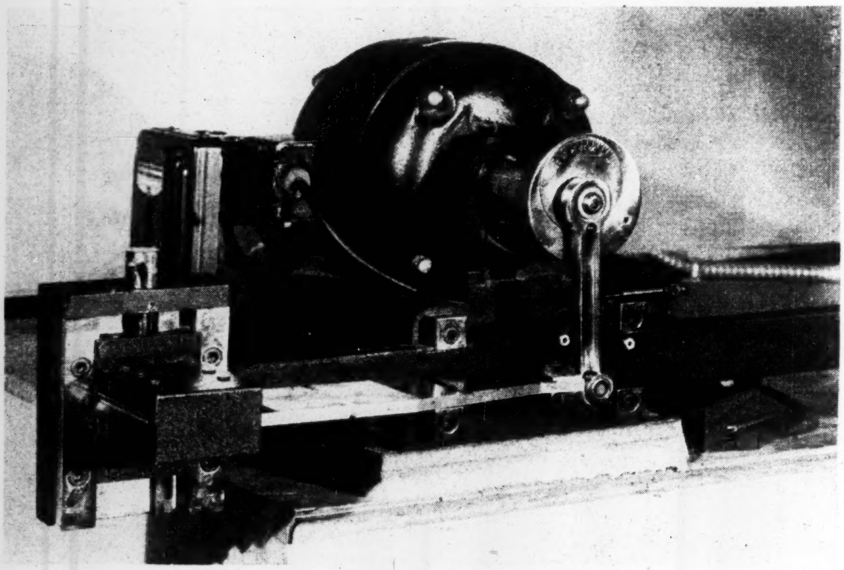


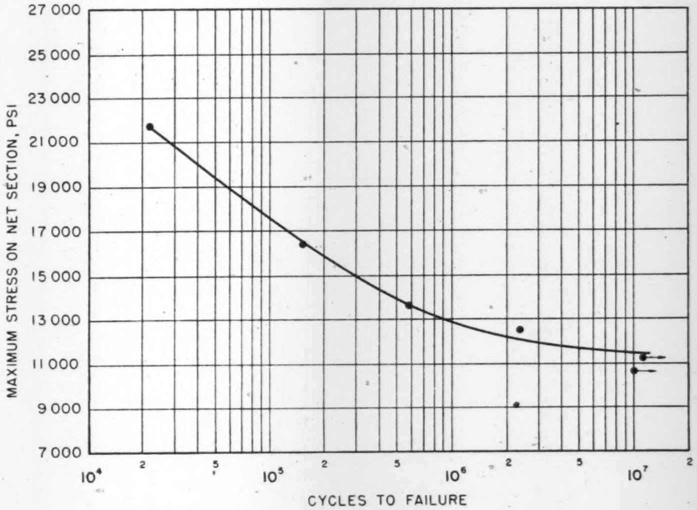
FIG. 2.—Plate-Bending Fatigue Machine.

Measuring Residual Stress:

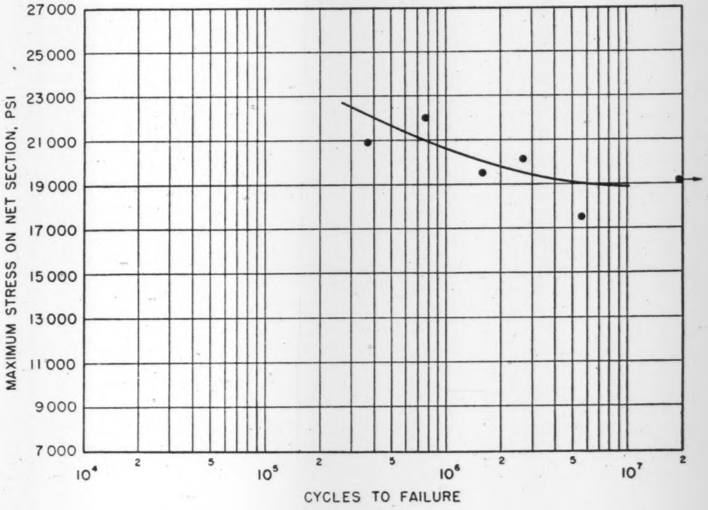
So far, X-ray diffraction is the only technique which enables one to measure the residual stress locally, and by being non-destructive is suitable for following any change in the magnitude of the residual stress during the fatigue testing. Lately, this technique has met with some suspicion because the X-ray stresses have been found to differ from the mechanically measured stresses beyond the yield point of the material in simple tension (4). Experiments performed on 61S - O* (5) have confirmed the existence of this discrepancy, but they have also indicated that the latter depended on the amount of plastic deformation. For deformations smaller than 1 per cent, the difference is within the experimental error of the X-ray technique, that is, within ± 1200 psi for the case of aluminum alloys. Therefore, insofar as the present work is concerned the technique may be used without any restrictions. The main features of this technique as applied to aluminum alloys have been described elswhere (6). Details pertaining to the present work are the same as those given previously (3).

Fatigue Testing:

The specimens were tested in the Krouse plate fatigue machine, Fig. 2, which bends a cantilever specimen by means of a crank and connecting rod to a predetermined value of deflection. Two ranges of load were employed: (*a*) reverse bending, in which the applied stress alternated between compression and tension of equal magnitude, and (*b*) pulsating bending, in which the applied stress varied between zero and tension. In the first case the mean applied stress was zero, in the second case it was half of the maximum applied stress. The alternating stress was obtained by setting the throw of the crank to reproduce a deflection of the specimen previously determined by dead weight calibration. The mean stress was set by adjusting the height of the butt of the specimen with respect to the crank by a screw movement on the vise. It, too, was determined by reproducing a dead weight deflection. The machine shut off automatically when the specimen failed and the attached counter read the number of cycles to failure.

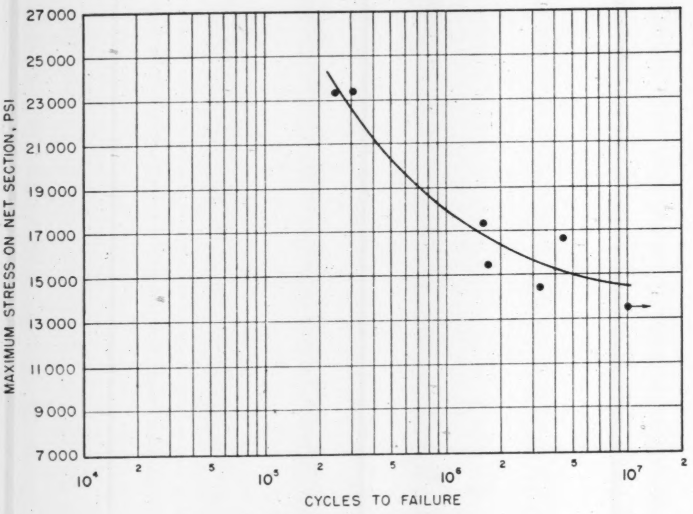


(*a*) Reverse bending.

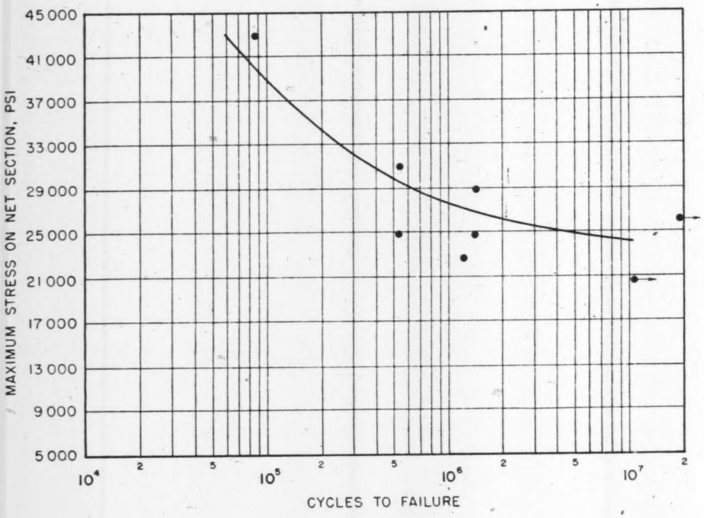


(*b*) Pulsating bending.

Fig. 3.—Unnotched 61S-O* Specimens.



(*a*) Reverse bending.



(*b*) Pulsating bending.

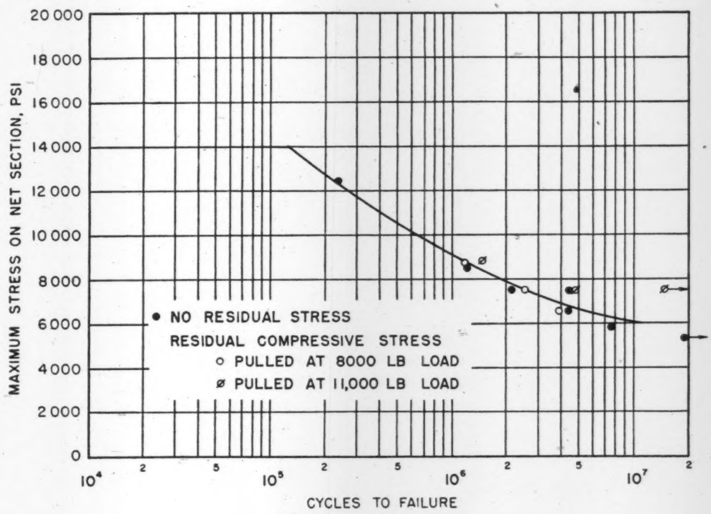
FIG. 4.—Unnotched 61S-T Specimens.

The probable error of the fatigue stress resulted mainly from the degree of reproducibility of the dead weight deflection in the course of testing, the error of measurement of the dimensions of specimens, and the variation of the modulus of elasticity. It was estimated to be of the order of 1 per cent, or ±100 psi on the average.

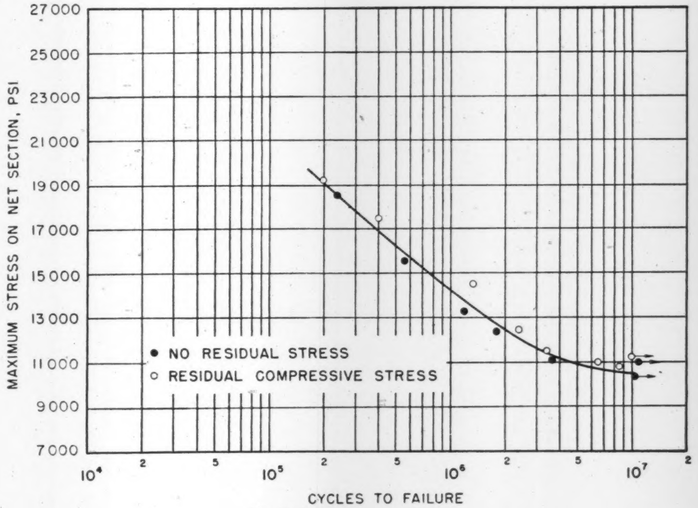
RESULTS

Fatigue Tests:

The results of the fatigue tests were represented by means of the S *versus* log N plots, where S ("the maximum stress on the net section") is the nominal fiber stress at the peak of the fatigue cycle computed by the usual bending formula with no regard to stress concentration, and N is the number of cycles for failure. Figures 3 and 4 contain results in reversed and pulsating bending of 61S-O* and 61S-T materials, respectively, without notches. Figure 5 refers to 61S-O* notched specimens with and without residual stress in both reversed and pulsating bending. The results of reversed bending were taken from a previous investigation (3). Figure 6 contains similar data for the 61S-T notched material. The results marked on the "No Residual Stress" curve by a special symbol[5] and all results plotted on the "Residual Tensile Stress" curve were obtained at a



(*a*) Reverse bending.



(*b*) Pulsating bending.

FIG. 5.—Notched 61S-O* Specimens.

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[5] Circle crossed with line.

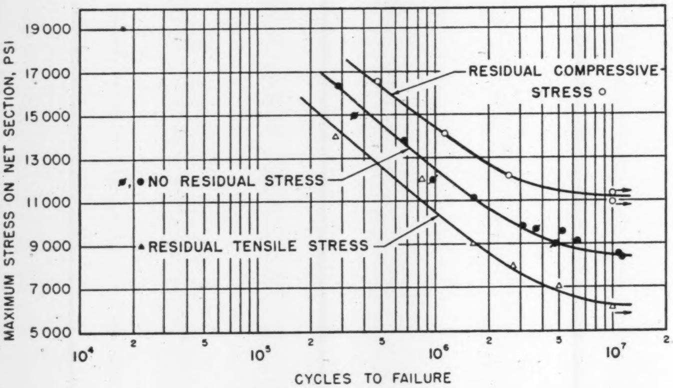
later date from a second batch of material.

Residual Stress:

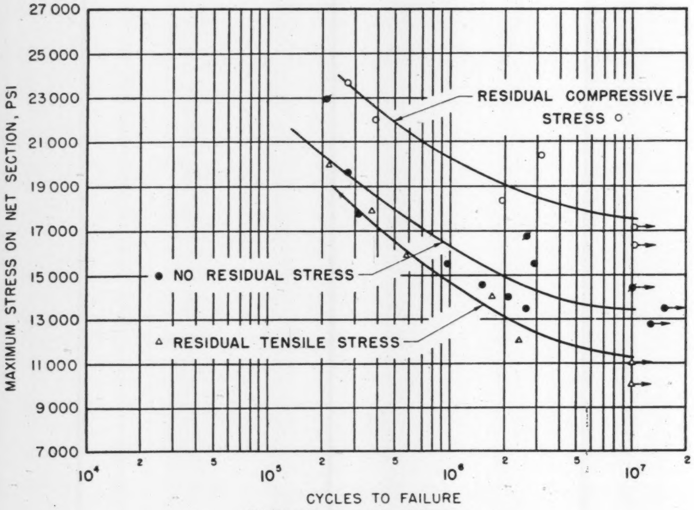
The determination of the residual stress was limited in the present work to a spot check at a point of the notched section located on the face of the specimen as close to the base of the notch as it was feasible (0.037 ± 0.005 in.). The recourse to a spot check measurement, rather than a detailed exploration, was justified on two scores:

1. In the 61S - O* material the residual stress was induced by approximately the same static load as in a previous work (3). A detailed exploration therefore was not deemed necessary, especially in view of the results obtained.

2. In the 61S - T material, the results showed considerable scattering presumably due to the preexisting stresses, as mentioned previously. Since the latter could not be removed by any known treatment without impairing the physical properties of the precipitation hard-



(*a*) Reverse bending.



(*b*) Pulsating bending.

FIG. 6.—Notched 61S-T Specimens.

Solid circles crossed with line refer to second batch of material ened alloy, there was little point in exploring the existing stress pattern in detail. It will be shown in the discussion that despite this scattering a reasonable explanation and prediction of the results can be made.

Column 4 of Table II contains the values of the residual compressive stress, R.S. induced by the static load stress, L.S. shown in column 2, prior to the fatigue testing. Column 8 gives the values of the residual stress left after, or induced by, the application of the fatigue stress, F.S., shown in column 6, at the end of 10 million cycles. Both the load and the fatigue stresses are the nominal stresses on the net section at the location of the notch. In columns 3, 5, 7, and 9, the above values are represented as fractions or multiples of the yield point of the material for the purpose of further discussion.

No residual tensile stress determinations were made because the grain size of the second batch of material which was used for the residual tensile series of Fig. 6, was too coarse for the X-ray technique employed (6).

TABLE II.—RESIDUAL STRESS AT 0.037 IN. FROM NOTCH.

	Before cycling				After 10 million cycles			
	Load Stress L.S.		Residual Stress R.S.		Fatigue Stress, F.S.		Residual Stress, R.S.	
	psi	L.S./Y.P.	psi	R.S./Y.P.	psi	F.S./Y.P.	psi	R.S./Y.P.
1	*2*	*3*	*4*	*5*	*6*	*7*	*8*	*9*
61S-O*	0		0		$6\ 000^b$	0.37	0	0
Y.P. =	14 500	0.89	$-9\ 000^a$	0.55	$6\ 000^b$	0.37	−3 500	0.20
16 300 psi	19 600	1.20	$-14\ 500^a$	0.87	$6\ 500^b$	0.40	−4 200	0.26
	13 000	0.80	− 6 000	0.37	$10\ 500^c$	0.65	−6 700	0.43
			− 7 300	0.45			−7 300	
	0		0		$10\ 500^c$	0.65	−6 000	0.37
61S-T	0		0		$8\ 500^b$	0.21		
Y.P. =			−17 000		$11\ 000^b$	0.27	−13 000	0.34
40 000 psi			−21 000		$17\ 500^c$	0.44	−11 000	0.27
	32 000	0.8	−19 700 Individual					
			−15 000 values					
			−13 500					
			−13 000					
	0		0		$13\ 500^c$	0.34		

[a] Average value from previous work (3).
[b] Reversed stress.
[c] Pulsating stress.

DISCUSSION OF THE RESULTS

Fatigue Strength of Unnotched and Notched Specimens:

From the results Figs. 3, 4, 5 and 6 fatigue strengths at 10^7 cycles are gathered in Table III, columns 2 and 3. Because of the greater statistical reliability the average rather than the minimum values at 10^7 cycles were taken

TABLE III.—FATIGUE STRENGTH OF UNNOTCHED AND NOTCHED SPECIMENS.

Material	Fatigue Strength, psi			Residual Stress at Notch, psi	K_t
	Unnotched	Notched			
		Measured	Predicted		
1	*2*	*3*	*4*	*5*	*6*
61S-O*	$11\ 500^a$	$6\ 000^a$		^{c}	1.90
Y.P. =		$6\ 000^a$		$-1\ 300^d$	
16 300 psi	$19\ 000^b$	$11\ 000^b$		$-9\ 300^c$	1.73
		$11\ 000^b$		$-10\ 600^d$	
61S-T					
Y.P. =	$14\ 500^a$	$8\ 500^a$		...	1.71
40 000 psi		$11\ 000^a$	$9\ 600^a$	−13 000	
		$6\ 000^a$	$6\ 400^a$	+24 000	
	$24\ 000^b$	$13\ 500^b$		...	1.70
		$17\ 500^b$	$15\ 700^b$	−11 000	
		$11\ 500^b$	$11\ 600^b$	+11 000	

[a] Reversed stress.
[b] Pulsating stress.
[c] Initially stress-free specimen.
[d] Initially prestressed specimen.

as the fatigue strength. It is seen that the scatter around the average values is greater for the pulsating than for the reversed stresses. The probable error of the reversed fatigue strength is estimated to be no more than ±500 psi on the average, while the probable error of the pulsating fatigue strength is not better than ±1000 psi on the average. The *S-N* curve for the 61S - O* alloy under pulsating load was limited to few points because the yield point in the outer fibers was exceeded even at the fatigue strength for 10^7 cycles.

Column 6 contains the value of the Fatigue Strength Reduction Factors defined by the ratio K_f =

$$\frac{\text{Fatigue Limit of Unnotched Specimen}}{\text{Fatigue Limit of Notched Specimen}}.$$

It is seen that K_f is about the same in all four cases and has an average value of 1.76. As shown previously (2) the notch has a stress concentration factor, $K_t = 2.5$. The fact that K_f is smaller than K_t is partially explained by the "size effect." (7) "Size effect" is used to describe the phenomenon of the lower permissible fatigue strength for large specimens as compared to that for smaller ones. Since the highly stressed material around the notch is very small, it shows a higher fatigue strength than the large uniformly stressed specimen used to obtain the properties of the material. As will be shown later, the ratio K_t/K_f has an important bearing on the influence of the residual stress on fatigue.

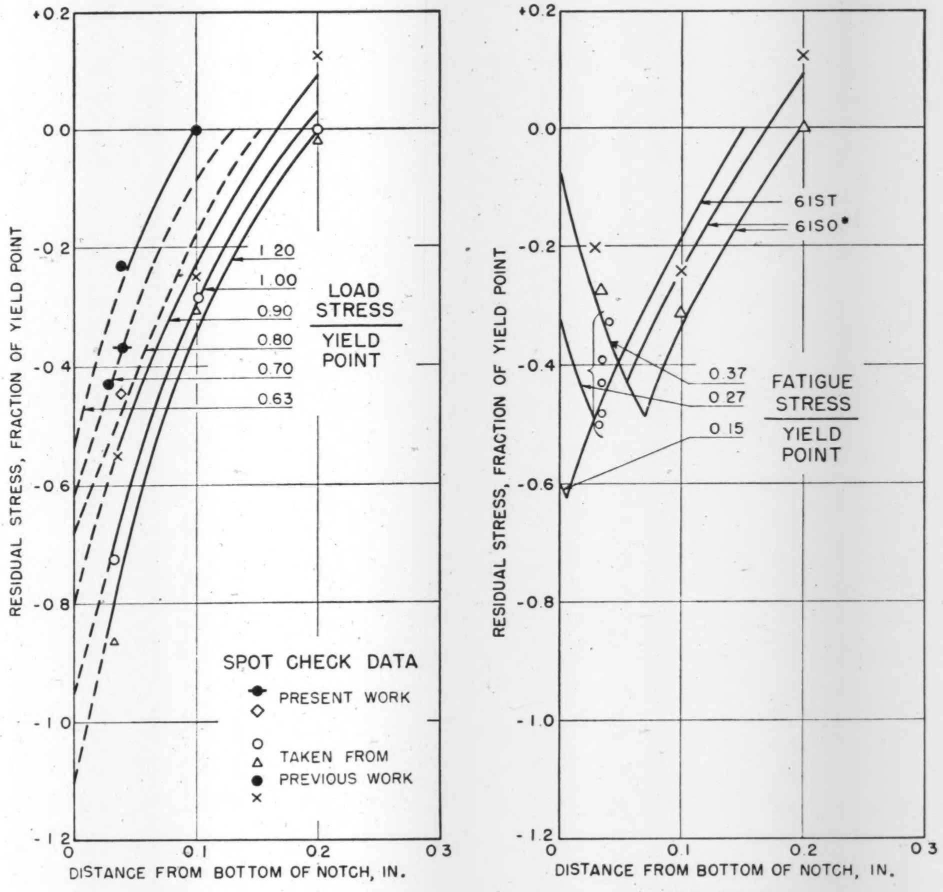
Influence of the Residual Stress on Fatigue:

Inspection of Fig. 5 shows that the residual stress induced prior to fatigue testing had practically no influence on the fatigue strength of the soft 61S - O* alloy. On the other hand, its influence on the fatigue strength of the hard 61S - T alloy was quite appreciable. It may be seen from Fig. 6 that by changing the residual stress from tension to compression an improvement of almost 100 per cent can be achieved in the fatigue strength of this alloy. In order to account for the different behavior of the two alloys attention is called to Table II.

Inspection of columns 4, 6, and 8 of Table II shows that in the case of the soft 61S - O* material the residual stress induced prior to cycling was reduced to about $\frac{1}{3}$ of its original value by the application of a reversed stress which was very nearly equal to the fatigue strength for 10^7 cycles. A corresponding value of the pulsating stress caused no change in the amount of the residual compressive stress which was set up prior to cycling. However, it did induce a residual compressive stress of its own of an almost equal magnitude in the initially stress-free specimen. On the other hand, the application of a cycling stress equal to the fatigue strength had practically no influence on the amount of the residual stress induced prior to cycling in the hard 61S - T alloy.

On the basis of the previous work (2) it may be asserted that the above situation did not develop gradually during cycling, but was established definitely after the first few cycles. In other words, the first few applications of the fatigue stress have substantially reduced the difference between the stress free and prestressed 61S - O* specimens, while they have left the situation in the 61S - T specimens very nearly unchanged.

In order to have a full understanding of the above phenomena one must examine the stress conditions at the point of initiation of the fatigue crack, that is, at the base of the notch. Since a direct measurement of the stress at this point was not feasible, an attempt has been made to secure this information indirectly. To effect this, all previous data regarding the amount of residual stress induced under various conditions of loading have been gathered in one diagram, Fig. 7(*a*). To eliminate the nature of the material the values of stress, residual as well as applied, have been plotted as multiples of the yield point. If small discrepancies in the stress-strain diagram of these materials slightly beyond the yield point are disregarded, this procedure is legitimate since the geometry of the notch has been kept very nearly the same in all specimens. The continuous lines have been drawn through the experimentally determined points. Broken lines represent linearly interpolated and extrapolated values. A measure of the reliability of the interpolation is supplied by the spot check data obtained in the present work on 61S-O* (columns 3 and 5, Table II). Because of the scatter of the data this criterion could not be applied to 61S-T. For clarity the range of this scatter is shown separately in Fig. 7(*b*) in which the curve marked 61S-T is the same as that marked 0.8 Y.P. in Fig. 7(*a*). It is seen that the scatter is in the direction



(*a*) Induced. (*b*) Relieved.

FIG. 7.—Residual Stress Pattern.

of lower values, as would have been expected from the presence of residual compression due to rolling. The curves marked 61S - O* are the same as those marked 0.9 and 1.2 Y.P. in Fig. 7(*a*).

The other curves in Fig. 7(*b*) give the amount of residual stress left after the first few applications of the fatigue stress. They have been computed following the procedure outlined in a previous work (**3**). This procedure is based on the following approximate relation:

$$R.S. + A.S. \leq Y.P. \quad \ldots\ldots\ldots (1)$$

where:

R.S. = the residual stress left after the application of the external (fatigue) load,

A.S. = the stress applied at the particular point of the notched section, and

Y.P. = the yield point of the material.

The two experimental points marked by a cross and a triangle on the 0.37 F.S./Y.P. curve are values taken from Table II, column 9, lines 2 and 3. The computation does not take into account the Bauschinger effect (**10**) the existence of biaxiality, nor does it make allowance for the redistribution of stress due to partial relief (**11**). The presence of a cusp in the curves is a result of these simplified assumptions. Nevertheless, the fact that predictions based on these curves are in reasonable agreement with experiments shows that the assumptions are not oversimplified, at least not for the present purpose.

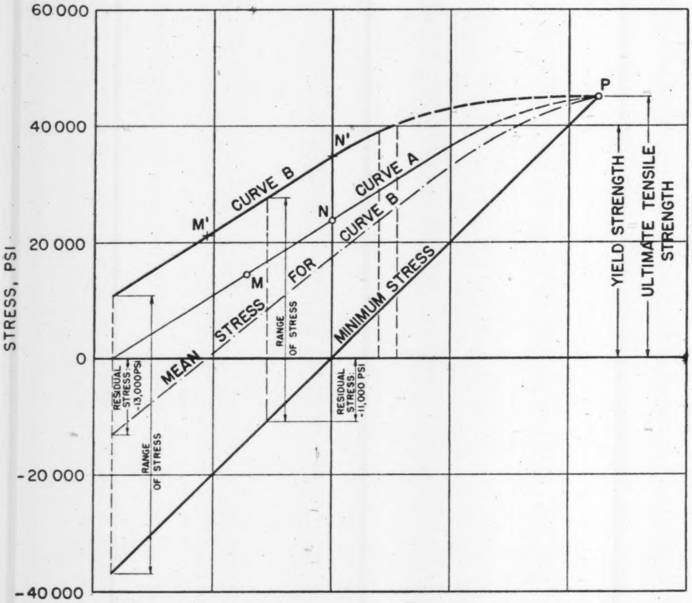


FIG. 8.—Modified Goodman Type Diagram of Unnotched and Notched 61S-T Material.

TABLE IV.—COMPUTATION OF RESIDUAL STRESS AT NOTCH.

Material	L.S./Y.P. or F.S./Y.P.	R.S./Y.P. Fig. 7(*a*)	F.S./Y.P.	R.S./Y.P. Fig. 7(*b*)	Residual Stress, psi
61S-O* Y.P. = 16 300 psi	0.89	0.8	0.37	0.08	−1 300
	0.67	0.57	...	...	−9 300
	0.80	0.65	...	...	−10 600
61S-T Y.P. = 40 000 psi	0.8	0.65	0.15	0.6	+24 000
	0.8	0.65	0.29	0.27	+11 000

With the help of Fig. 7 one can compute the amount of residual stress induced or left at the base of the notch after the application of a given value of fatigue stress. This amount is indicated in column 5, Table III.[6]

It will now be shown that with the above information at hand a reasonable prediction can be made as to the influence of the residual stress on fatigue.

Prediction of Fatigue Results in 61S - T:

The case of the hard, 61S - T alloy will be examined first. If the residual stress is superimposed on the fatigue stress, the limits between which the fatigue stress oscillates are shifted toward higher or lower values, but the range of stress is not affected. Thus, the residual stress modifies only the value of the mean stress.

On this basis, it should be possible to account for its influence on the fatigue strength if the dependence of the safe range of stress on the mean stress is known. This dependence has been determined as follows by making use of a modified Goodman type diagram (8). Curve *A*, Fig. 8, is the locus of the fatigue strengths for the notch-free material. It has been constructed from three experimental points: (*1*) the static tensile strength, point *P*, for which the safe range has been taken as zero (*2*) the pulsating fatigue strength, point *N*, for which the safe range is equal to the value of the fatigue strength, and (*3*) the reversed fatigue strength, point *M*, for which the safe range is equal to twice the value of the fatigue strength. A modified Goodman diagram for 17S - T aluminum alloy, determined by the Research Laboratories of the Aluminum Company of America (8), indicated the manner in which the curve might be interpolated between and extrapolated from the three experimental data. The safe range of stress for the notch-free material is given by the vertical distance between the lines marked "Minimum Stress" and "Curve *A*."

For the notched specimens the fatigue strength at the base of the notch is higher because of the size effect discussed previously. It is easily seen that the increase is in the ratio of

$$\frac{\text{Stress Concentration} \times \text{Fatigue Limit of Notched Specimen}}{\text{Fatigue Limit of Unnotched Specimen}} = \frac{K_t}{K_f} = 1.47$$

for both the pulsating and reversed fatigue strengths. The points *M′* and *N′* of the curve *B* are thus obtained. The curve itself has been drawn through these points and point *P* by following the trend of curve *A*. It is appreciated that the part of the curve between *N′* and *P* is highly speculative.

The safe range of stress for the small volume of material at the base of the notch thus is given by the vertical intercepts between the lines marked "Minimum Stress" and "Curve *B*."

If now the values of the residual stress listed in column 5, Table III, are superimposed on the mean stress, Fig. 8, the safe range of stress can be read directly from the plot. It should be noted that when the applied stress is reversed, the residual stress is plotted on the mean stress curve, but when the applied stress is pulsating the residual stress is plotted on the minimum stress line. As an example, the case of the notched 61S-T with residual compressive stress tested under reversed and pulsating loading is marked on the diagram. By plotting the residual stress of −13,000 psi on the mean stress line, it is seen that the predicted safe range of stress is 48,000 psi at the base of the notch, or $\pm \frac{24{,}000}{K_t} = \pm 9600$ psi for the notched reversed fatigue strength, since $K_t = 2.5$. The experimental value is ±11,000 psi. In like manner, by plotting the residual stress of −11,000 psi on the minimum stress line the predicted safe range of stress is 39,000 psi or $\frac{39{,}000}{2.5} =$ 15,700 psi for the notched pulsating fatigue strength. The experimental value is 17,500 psi. In both cases the measured and predicted values are within the combined probable error affecting the measurement of the residual stress and the determination of the fatigue strength. By proceeding similarly for the tensile residual stresses, the predicted values of ±6400 psi and 11,600 psi (column 4, Table III) are obtained. It is seen that the agreement between the predicted and measured values here is very satisfactory.[7]

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[6] Because of the scatter of the residual compressive stresses in 61S-T the values indicated in column 5 (−13 000 and −11 000) have not been computed but are those measured at 0.037 in. from the notch. The other values are computed in Table IV.

Prediction of Fatigue Results in 61S-O:*

From column 5, Table III, it is seen that the amount of the residual stress computed at the base of the notch for the notched fatigue strength indicated is about the same in the initially stress-free and the initially prestressed specimens. More specifically, the reversed fatigue stress has reduced the residual stress to less than 2000 psi compression in the prestressed specimen, while the pulsating fatigue stress has induced a residual stress of its own of 6000 psi compression in the initial stress-free specimen. In the final analysis, the two series of specimens which were originally with and without residual stress differ from each other by an amount of residual stress that is less than 0.08 Y.P. From the trend of the Goodman diagram, it may be inferred that this amount is too small to influence the safe range of stress significantly, which is in agreement with the experiments.

Criterion for Residual Stress Influence:

Since reasonable predictions of the fatigue results appear possible, one may attempt to formulate a criterion governing the influence of the residual stress on fatigue. The residual stress, as mentioned previously, modifies only the mean stress. Therefore, the residual stress is of influence to the same extent as the safe range of fatigue stress is a function of the mean stress. But even then, two additional conditions must be met depending on whether the residual stress has or has not the same sign as the fatigue stress.

1. When the residual stress has the same sign as the fatigue stress, the maximum amount of the residual stress which is left after a few applications of the fatigue stress is governed by the relation given previously:

$$R.S. + A.S. \leq Y.P. \quad \text{.........} (2)$$

where:

$A.S.$ = the stress applied at the base of the notch.

Using the previously defined coefficients K_t and K_f we can rewrite Eq 1 to read

$$R.S. + \frac{K_t}{K_f} \times F.S. \leq Y.P. \quad \text{......} (3)$$

where:

$F.S.$ = the fatigue strength of the unnotched material.

This formula applies when the residual stress has the same sign as the upper limit of the fatigue stress range.

When the residual stress has the same sign as the lower limit of the fatigue stress range, the relation:

$$R.S. + \frac{K_t \times R}{K_f} \times F.S. \leq Y.P.$$

must be satisfied. R is the ratio of the lower to the upper limit of fatigue stress range.

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[7] Instead of Fig. 7(*b*) one can use directly the Goodman diagram to obtain simultaneously the values of the fatigue strength and the corresponding residual stress by virtue of the relation (1). Obviously, the beneficial as well as detrimental effect of the residual stress cannot go beyond the limits imposed by this relation.

Obviously, a necessary condition for the residual stress to be of influence is that there be some left after the application of the fatigue stress. This condition requires that

$$\frac{Y.P.}{F.S.} > \frac{K_t}{K_f} \qquad \text{(4)}$$

or

$$\frac{Y.P.}{F.S.} > \frac{K_t \times R}{K_f} \qquad \text{(5)}$$

whichever is applicable.

As an example, $\frac{K_t}{K_f} = 1.47$ for both 61S-T and 61S-O*. But in reverse bending $\frac{Y.P.}{F.S.}$ is 2.65 for the first material and 1.42 only for the second. By virtue of Eq 4 or 5 the residual stress has an influence on the fatigue results in the first case, but has none in the second in agreement with the experiments.

2. When the residual stress has the opposite sign to that of the fatigue stress, it remains unaffected by the latter, but it can still be without influence on the fatigue results, if the service loading itself induces substantially the same amount of residual stress in the previously stress-free specimen. One way this can happen is when the fatigue stress has about the same value as the load stress used for inducing the residual stress (pulsating tests data of 61S-O*.

No accurate mathematical prediction of the amount of the residual stress induced is available, so the second condition cannot be expressed quantitatively as yet. The effect of the experimentally determined residual stress, however, is amenable to quantitative analysis as has been shown in this discussion, but even here not enough data are available to completely justify the assumptions which have been made. Other values of K_t and K_f also must be investigated. This problem has been reserved for future studies.

CONCLUSIONS

The following conclusions appear justified:

1. In the absence of any other treatment, residual stress alone induced at the base of the notch can have a beneficial as well as detrimental effect on the fatigue strength depending on sign. But it can also have no significant influence. Thus, the 61S Aluminum Alloy in the precipitation hardened 61S-T6 condition has shown an improvement of almost 100 per cent when the residual stress was changed from tension to compression. The same alloy in a softened condition was unaffected by this treatment.

2. The reason for the above discrepancy lies in the extent of the change which the first few cycles have brought about in the originally induced residual stress. While little or no change was found in the case of the hard 61S-T alloy, the changes produced in the soft 61S alloy in effect nullified any significant difference between the prestressed and stress-free specimens.

3. Whether the residual stress will or will not be of influence appears to depend on whether the ratio of the yield point to the fatigue limit of the material is greater or smaller than the ratio of the stress concentration factor to the fatigue strength reduction factor. The amount of the residual stress which the fatigue stress can set up in the originally stress free specimens also is important. Further studies are necessary to account quantitatively for this factor.

4. A reasonable prediction of the effect of residual stress on fatigue appears possible on the basis of a modified Goodman diagram.

5. In the absence of any established criterion the X-ray stress technique within its limitations can be used as a criterion for the residual stress influence on fatigue.

Acknowledgment:

The authors are indebted to the Department of Engineering, the Research Grant Committee of the University of California, Los Angeles, and the Levine Engineering Fund for the facilities and funds which enabled them to carry out this work. They also wish to express their appreciation to P. V. Faragher of the Aluminum Company of America for supplying them with the necessary material.

REFERENCES

(1) O. J. Horger, "Residual Stresses," *Handbook of Experimental Stress Analysis*, John Wiley and Sons, Inc., New York, N. Y. 1950. See also:
G. Forrest, *Journal*, Inst. Metals, Vol. 72, pp. 1–7 (1946).
R. Schmidt, *Luftwissen*, Vol. 9, pp. 263–67 (1942).

(2) J. T. Norton, D. Rosenthal, and S. B. Maloof. *The Welding Journal*, Vol. 25, Research Supplement, November, 1946, pp. 729-s to 736-s.

(3) D. Rosenthal, G. Sines, and G. Zizicas, *The Welding Journal*, Vol. 28, Research Supplement, March, 1949, pp. 98-s to 104-s.

(4) S. L. Smith and W. A. Wood, *Proceedings*, Royal Soc., London, Vol. A182, p. 404 (1944).

(5) J. P. Frankel. Ph.D. Thesis (1951). Department of Engineering, University of California, Los Angeles.

(6) L. Frommer and E. G. Lloyd, *Journal*, Inst. Metals, Vol. LXX, pp. 91–112 (1944).

(7) R. E. Peterson, "Methods of Correlating Data from Fatigue Tests of Stress Concentration Specimens," *Stephen Timoshenko Anniversary Volume*. Macmillan, pp. 179–183 (1938).

(8) "Prevention of the Fatigue of Metals," Battelle Memorial Institute, John Wiley, New York, N. Y., p. 134 (1941).

(9) D. Rosenthal, R. Hultgren, and D. I. Martin, "The Use of X-rays for the Determination of Stresses in Ship Plate," *N.D.R.C. Research Project*, N.R.C. 74, (1944).

(10) F. Seitz, "The Physics of Metals," McGraw-Hill, Book Co., Inc., New York, N. Y. (1940).

(11) J. T. Norton and D. Rosenthal, *Journal*, Am. Welding Soc., Vol. 22, Research Supplement, pp. 63–78 (1943).

DISCUSSION

CHAIRMAN T. J. DOLAN.[1]—In Fig. 6(*b*) of the paper showing the effect of pulsating bending on specimens having a residual stress, the authors have shown that four or five of these specimens were loaded to nominal stresses above 18,000 psi. Based on their theoretical stress concentration factor of 2.5, one arrives at a theoretical stress at the notch of about 45,000 psi which I believe is above the yield point. Do the authors know of any reason why these specimens did not yield to relieve the residual stresses and thus fall on the curve for specimens having no residual stress as happened in the case of the 61S-O* specimens?

MR. R. L. TEMPLIN.[2]—In some of the fatigue tests made at the Aluminum Research Laboratories, using the R. R. Moore type of specimens and a very sharp (radius less than 0.0002 in.) 60 deg. V-type notch, minimum diameter 0.330 in. and maximum diameter 0.480 in., and a still higher strength aluminum alloy than those used by the authors, we found, as expected and as shown by the authors, that axially prestressing the specimens in tension to a rather high value, previous to fatigue testing, gave a residual compression stress at the bottom of the notch which resulted in considerably higher fatigue values, as normally determined, than the specimens not so prestressed. Similar specimens of the same material initially prestressed in compression to a corresponding value, so as to leave a residual tensile stress at the bottom of the notch, gave appreciably lower fatigue values than the specimens which were not prestressed.

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[1] Research Professor of Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

[2] Assistant Director of Research and Chief Engineer of Tests, Aluminum Company of America, New Kensington Pa.

The specific material referred to was 75S-T6 bar stock having a tensile strength of 83,800 psi, a yield strength (0.2 per cent offset) of 72,600 psi, and an elongation in 2 in. of 14 per cent. When tested in tension using a notched specimen similar to and of the same size as the R. R. Moore type fatigue specimen, this material had an apparent tensile strength of 95,300 psi (total load on the specimen divided by the minimum cross-sectional area). For prestressing the notched fatigue specimens, a nominal stress corresponding to 88 per cent of this value was used, namely, 84,000 psi, both in tension and in compression.

The notched fatigue specimens which were not prestressed had a fatigue strength at 10^7 cycles of 12,000 psi, whereas those prestressed in tension but having a residual compressive stress at the root of the notch, had a fatigue strength at the same number of cycles of 21,000 psi. Those prestressed in compression so as to give a resulting tensile stress at the root of the notch had a fatigue strength of 4000 psi at 10^7 cycles. The material used in these tests had a fatigue strength at 10^7 cycles, using the regular R. R. Moore specimens, of 29,500 psi. From these results it may be readily shown that the K_f values are 2.46 for the notched specimen without prestressing, 1.40 for the notched specimen in which the residual stress is compression, and 7.37 for the notched specimen in which the residual stress is tension.

These results would appear to agree at least qualitatively with those obtained by the authors. Certainly 75S-T6 material has a yield strength appreciably above its normal fatigue strength at 10^7 cycles.

There is another point in connection with fatigue tests, not only those reported by the authors but in the previous papers, that I have not heard mentioned specifically and that has a bearing on the scatter problem in fatigue results. We go to great lengths in careful preparation of surface, by machining, and polishing to obtain the conditions desired, then put the specimens in a fatigue machine and run them for long periods of time. In the old rotating-beam type of fatigue machine, running at a speed of about 1750 rpm, it required about six months' time, at or near the lowest stress at which the specimens were run, before the test was considered finished. Depending on the material, I think it is reasonable to expect effects of atmospheric conditions on the fatigue results obtained, depending on the vicissitudes of humidity and whatnot that obtain. This is not a new observation; it stems back to work done in England many years ago when specimens were run in vacuo, and also I believe, protected by mineral oil.

In the interest of cutting down some of the scatter—perhaps not all of it, by any means—in our own fatigue tests it has been customary for the past twenty-odd years to apply some form of protection against such atmospheric conditions as might affect the fatigue results.

There are available today good commercial waxes that serve very well in helping protect fatigue specimens against the effects of humidity and perhaps other atmospheric conditions that may tend to give us, without our wanting to have it, a corrosion-modified or mild form of corrosion-fatigue test. This protection, we believe, helps decrease the width of the scatter band but does not eliminate the scatter entirely by any means.

MESSRS. D. ROSENTHAL AND G. H. SINES (*authors' closure*).—In reply to the question by Mr. Dolan, we should like to point out that the effect of the pulsating load above the yield point is quite different for the cases of residual compression and residual tension. In the first instance, the amount of residual compressive stress will remain unchanged as long as the nominal pulsating stress is smaller than the load stress which was used initially to induce this amount and which was equal to 32,000 psi. In the second instance the residual tensile stress will be totally relieved as soon as the nominal pulsating stress reaches the value of 18,000 psi indicated by Mr. Dolan. This can be shown as follows:

1. It will be recalled that the residual compressive stress was originally induced by applying a nominal tensile stress of 32,000 psi. Since the pulsating load is also tensile, it cannot relieve the residual compressive stress at the notch. Nor can it increase its amount, for the maximum pulsating stress employed in the tests is well below the value of 32,000 psi used for setting up the residual stress initially.

2. In the case of the residual tensile stress, the applied and residual stresses are of the same sign. Hence, the criterion, Eq 2 is valid, and by this criterion a nominal stress of 18,000 psi, resulting in an applied stress of 18,000 × 2.5 = 45,000 psi at the notch, will relieve the residual stress at the notch. As a result, we should expect the "residual compressive stress" curve to be above the "no residual stress curve" at all applied stresses and the "residual tensile stress" curve to merge into the "no residual stress curve" beyond 18,000 psi. This trend actually can be observed in Fig. 6(*b*) within the scatter of the experimental results.

We are indebted to Mr. Templin for the inclusion of the data on 75S-T6 in the present discussion. It seems reasonable to assume that the effect of the residual stress on fatigue can be made much greater by using sharper notches, but the measurement of stress at the bottom of these notches becomes so difficult and uncertain as to preclude any quantitative predictions. Regarding the technique of setting up the residual stress by preloading we, of course, do not presume to be the first ones to use this technique. In the reference (1) to our paper, we have mentioned the name of Mr. G. Forrest who made use of it in a series of tests in which the effect of residual stress on fatigue was conclusively shown. Also, J. Almen has presented data in his articles bearing on the same subject. However, in neither of the cases considered was there any attempt made to control the effect of cold working, nor was the amount of one residual stress measured and followed during testing. We believe that without these two precautions it would be very difficult to make quantitative predictions regarding the effect of residual stress on fatigue. In our tests, an attempt has been made to minimize the effect of cold working so that this factor could be neglected. Future tests will have to deal with the combined effect of cold working and residual stress.

With reference to Mr. Templin's remark about the effect of humidity on fatigue results, perhaps the blame for the scatter of some of our results should be put elsewhere, since the Los Angeles area enjoys a low and rather constant degree of humidity.

In conclusion, we should like to thank the discussors for their contributions.

DAMPING, ELASTICITY, AND FATIGUE PROPERTIES OF TEMPERATURE-RESISTANT MATERIALS*

By B. J. Lazan[1] and L. J. Demer[1]

Synopsis

The damping, elasticity, and fatigue properties of several temperature-resistant materials were investigated in rotating cantilever-beam testing equipment. The room and elevated temperature tests were designed to reveal changes in damping energy and dynamic modulus of elasticity during constant cyclic stress fatigue tests at engineering stress levels. Usual *S-N* fatigue curves are presented in addition to a series of new diagrams designed to show the effects of both stress magnitude and stress history on the damping and elasticity properties. Two methods for comparing the damping energies of a group of materials are offered and the merits of each discussed. Diagrams are also presented to facilitate comparison of the elasticity properties among materials tested at a given temperature.

Most of the data which have been reported in the literature on the damping and dynamic modulus of elasticity properties of metallic engineering materials were secured either under conditions of very low constant stress or else under conditions of varying stress magnitude and stress history. To be of greatest usefulness in the development and selection of materials as well as for machine-design purposes, the data should be obtained from tests conducted under constant stress conditions at levels of practical engineering importance. Furthermore, the important variable of stress history should be carefully controlled.

The work reported in this paper is part of a long-range test program of the above type dealing with the damping, elasticity, and fatigue properties of materials at engineering stress levels. It follows an intensive investigation of the properties of mild steel (**1**)[2] and includes tests on a number of temperature-resistant materials carried out both at room and elevated temperatures.

Objectives and Engineering Importance

The increasingly rapid pace of development in gas turbines, propulsion engines, and other high temperature applications has emphasized the importance of a better understanding of the mechanical properties of temperature-resistant materials. Data on the elevated temperature properties under dynamic forces are particularly inadequate. This fact, combined with uncertainty as to what constitutes service, makes design engineering in this field particularly difficult. As a consequence, there have been many cases of underdesign with resultant service failure, and no doubt there exist the less spectacular but equally costly errors of overdesign with resultant inefficiency due to failure to utilize fully the properties of materials. In many cases this uncertainty is a result of the difficulty in interpreting laboratory data and correlating these data with field requirements.

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* Presented at the Fifty-fourth Annual Meeting of the Society, June 18–22, 1951.

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[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 646.

One of the least understood of the dynamic mechanical properties is damping. Not only are there practically no engineering data on the damping properties of temperature-resistant materials at elevated temperatures, but the interpretation of these few data is generally quite difficult. Consequently, the significance of damping and its utilization by the engineer for design and failure analyses purposes is generally ignored.

The dynamic modulus of elasticity and its change under sustained cyclic stress is another property of importance to design engineers. An attempt is made, therefore, to present in this paper not only new data on the damping, elastic modulus, and fatigue properties of materials but also to discuss the interpretation, significance, and interrelationships of these data.

The experimental data presented in this paper were procured with newly developed rotating-beam equipment which enables continuous measurement of the damping energy and dynamic modulus of elasticity during the course of a rotating-bending fatigue test. Engineering data on the following three dynamic properties of engineering materials are therefore presented and analyzed:

1. Damping energy as a function of stress magnitude and stress history,
2. Dynamic modulus of elasticity as a function of stress magnitude and stress history, and
3. Rotating-bending fatigue strength.

The general engineering importance of these three dynamic properties has been discussed briefly (1, 2, 3). However, these properties assume special significance, as discussed below, when concerned with temperature-resistant materials used in turbine blades and other heat engine parts.

Damping Energy:

The damping properties of a material may be defined in several different ways (1, 3). In this paper, damping is specified, in terms of damping energy, in units of inch-pounds of energy absorbed by 1 cu in. of the metal during one complete cycle of vibration. This energy is represented by the area within the stress-strain hysteresis loop as discussed in previous publications (1, 6). The engineering importance of damping energy in general machine design, and consequently in the development, selection, and processing of engineering materials, has been discussed (3).

Of the several advantages of high damping energy, probably the most important in turbine and heat engine design is that related to the limitation of stress due to near-resonant vibrations. In modern turbines and other high-speed heat engines, near-resonant vibrations are generally rather difficult to avoid. It is commonly believed that the increased and uncontrolled stress resulting from these near-resonant vibrations constitute one of the more common causes for service failure (5).

The amplification in stress resulting from near-resonant vibrations is reciprocally dependent on the total effective damping capacity of the entire *system* partaking in the vibration. This relationship may be expressed as follows:

Alternating stress at resonance
= (alternating stress applied externally) $\times$ (K_r/D_0) . . . . . (1)
= (alternating stress applied externally) $\times$ A_r . . . . (2)

where:

D_0 = damping energy absorbed by *system* in units of in-lb per cycle of vibration,

K_r = constant depending on the system, and

A_r = resonance amplification factor, unitless = a direct measure of the increase in stress attributable to the resonant condition of a vibration.

The above discussion is in terms of the behavior of a "system." The damping of a system includes not only (*a*) the *internal* hysteresis damping of the material making up the system, but also (*b*) the *external* damping of joints, air friction, dash pots, and other units attached to increase mechanical damping. The relative importance of *internal* damping as compared with *external* damping depends, of course, on the system. It is generally believed, although there is practically no substantiating data, that in most cases external damping is larger than internal damping. However, there is little doubt that in some systems the source of practically all the damping is internal hysteresis. A testing program now in progress at the University of Minnesota is designed to evaluate the relative importance of the various components making up the total damping in different types of systems. However, this paper is concerned entirely with internal hysteresis damping and excludes consideration of extraneous or external damping. This should not be considered a serious limitation since the study of hysteresis damping constitutes a necessary first step. Furthermore, even in cases where there is considerable extraneous damping, high internal damping is still very helpful in limiting near-resonant vibrations.

Different materials possess widely different damping capacity. In some materials at engineering stress levels, the resonance amplification factor may be as high as 1000; in others, as low as 5. Most practical cases, however, lie in the range from 5 to 100 at stresses near the fatigue limit.

In choosing a material for a part which may receive near-resonance vibration in service, it is usually unjustified to use fatigue strength as the main criterion for judgment; the damping must also be considered. For example, Föppl (7) has stated that "the endurance of (airplane propeller) blades depends far more on damping capacity of the material than its fatigue strength." Similarly, overhead cables and aircraft parts made of a low-strength, high-damping material may outlast others made of a high-strength, low-damping material. This relationship of the fatigue and damping properties to the destructive effect of resonant vibration is further discussed in connection with the experimental data presented later.

Dynamic Modulus of Elasticity:

The dynamic modulus of elasticity is defined (1, 6) as the slope of the secant line between the zero and the maximum stress-strain point in the hysteresis loop present during cyclic stress. Stated differently, it is the ratio of the maximum stress to the maximum strain during the cycle. The general significance of dynamic modulus and reasons for its variation during sustained cyclic stress have been discussed elsewhere (1, 4, 6). In so far as turbine blades and other high-temperature applications are concerned, dynamic modulus is considered primarily because of its direct effect on:

(*a*) the position of the resonant frequency spectrum of a part with respect to its operating speeds and

(*b*) the amplitude of vibration of a part, either near to, or remote from, resonance under a given exciting force.

In connection with the first of these considerations, it is generally desirable to design a part so that the lowest critical frequency of its resonant frequency spectrum is above the operating speed of the machine or its serious harmonics. If this cannot be done, which is usually the case in turbine blades, etc., the part is designed so that the operating speed lies between two of the lower critical speeds of the part. In such cases only transient resonant vibrations during "speed-up" need generally be considered rather than the more destructive steady state resonant vibrations. Needless to say, any change in dynamic modulus of elasticity such as occurs during sustained cyclic stress will shift the resonant frequency spectrum and change the "spread" between the operating frequency and one of the resonances. The resultant near-resonant vibrations that may be so developed during prolonged service may, of course, be quite destructive. The importance of knowing dynamic modulus as a function of not only stress but also stress history is thus apparent.

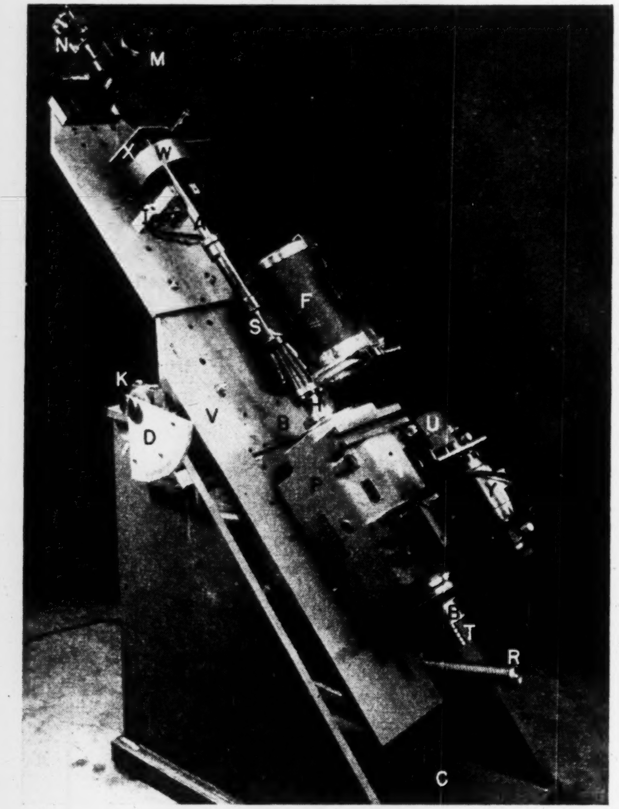

FIG. 1.—Damping, Elasticity and Fatigue Machine for High-Temperature Testing.

As in the case of damping, the resonant frequencies for a given design depend not only on the material, but also on the joints and other structural factors. However, as mentioned previously, a knowledge of the behavior of the material is a necessary first step.

Fatigue Strength:

The implications of fatigue stress are relatively well known and need not be reviewed at this time. It should be emphasized, however, that service failure records (5) indicate that fatigue stress in turbine blades constitutes a major cause of breakdown. In this connection, the relationship of magnitude of fatigue stress to the damping properties and proximity to resonance as determined by the dynamic modulus should be reemphasized.

It is thus apparent from the above discussion of damping, dynamic modulus, and fatigue strength that these three properties are not only important individually, but also their interrelationships have considerable engineering significance.

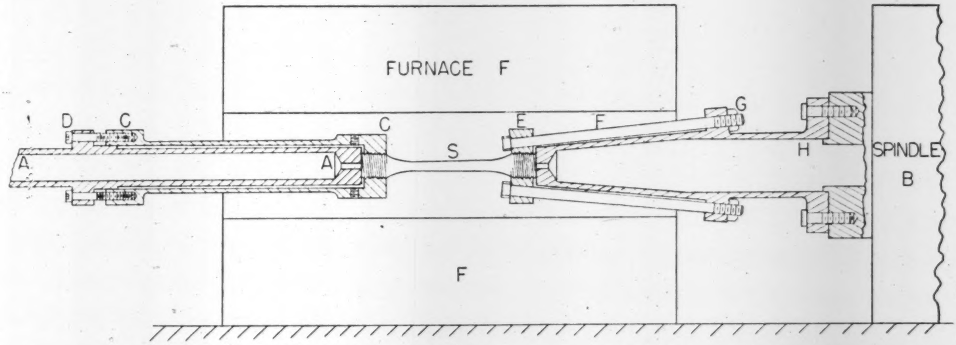

FIG. 2.—Schematic Diagram of Elevated Temperature Gripping System for Rotating-Bending Machine.

TESTING EQUIPMENT, PROCEDURES, AND METHODS OF COMPUTING DATA

Recently developed (1, 4) rotating cantilever-beam equipment was used in this program to procure damping and dynamic modulus data as a function of stress magnitude and stress history. Two of the room temperature models of this machine were converted for elevated temperature testing as shown in Fig. 1. A heavy base *C* with vertical sides *G* supports pillow blocks *K* through which passes a shaft attached to table *V* allowing the table to be tilted about a horizontal axis. The tiltable table supports spindle *B-B* which is mounted in accurate bearings contained in housing *P*. Electric motor *Y* rotates the spindle through a belt drive and also operates a revolution counter *U*. An extension arm-specimen-loading weight assembly *H-S-A-W* is attached to the spindle and rotates with it. During this rotation the horizontal and "vertical" location of a target in the top end of arm *A* may be measured with micrometer slide and microscope assembly *M-N*. A damper disk attached to the upper end of arm *A* through a rounded brass bushing rides on damper plate *X* and eliminates high frequency vibrations of the arm which would make accurate reading of the target position difficult. The damper plate lends no support to the arm and weight assembly. A bracket *I* serves to catch the arm and weight assembly *A-W*, and a microswitch attached to this bracket interrupts the power to the driving motor and furnace when the specimen *S* fractures at the end of a fatigue test. Furnace *F*, when in its proper location, surrounds the specimen and parts of the adjoining extension arms. Thermocouples are attached to the specimen for indication and control purposes. Leads from the thermocouples are directed through the hollow grips and hollow spindle *B*, and by use of slip rings *T* and a brush assembly (not shown) are connected to a temperature controller. The reversed bending stress to which the test specimen *S* is subjected is proportional to the sine of the angle between table *V* and the vertical direction. This angle, indicated on scale *D*, is steplessly adjustable by means of screw *R* which is mounted on the base and attached to the lower end of the table.

The method of gripping specimen *S* within furnace *F* is shown in Fig. 2. Extension arm *H*, attached to spindle *B*, is made hollow so as to minimize heat transfer from the furnace to the spindle bearings. The bottom end of the specimen is held rigidly against arm *H* by nut *E* and studs *F*, the pre-load in the assembly being large so as to avoid relative motion within the grip with consequential fretting and energy loss. The threaded left end of the specimen is similarly held against loading arm *A* by sleeve-nut combination *C*, preloaded by means of screws *D*. The arm-specimen assembly *S-A* is carefully lined up on dead centers by preferentially tightening the various holding and adjusting screws prior to attachment to the spindle. The assembly is then attached to the spindle extension arm *H* at room temperature and adjusted to run true by means of preferentially tightening nuts *G* on studs *F*. The furnace is then placed in position down over the loading arm and the weight *W* is mounted on the arm.

Even though the specimen-arm assembly is adjusted to run true on the spindle at room temperature, it is frequently found that the run-out of arm *A* and its measuring target may become significant after the assembly reaches the testing temperature. In such cases, nuts *G* are again preferentially tightened after the temperature has stabilized to make the measuring target again run true.

The gripping method employed permits use of relatively simple threaded specimens. Considerable leeway in accuracy is allowed in the threaded ends since the alignment adjusting system described above compensates for thread errors. Furthermore, preloading the threaded connections eliminates backlash and the accompanying fretting corrosion. Consequently, the "frozen" joint condition, which frequently occurs in fatigue testing at elevated temperatures, is practically eliminated.

For the measurement and control of specimen temperature, leads from three thermocouples spaced at the top, middle, and bottom of the specimen test length were carried through chromel and alumel slip rings and brushes to the temperature controller and indicating potentiometer. The maximum variation in temperature between the bottom and top of a specimen and the variation in temperature at a station during a complete test were in most cases about ± 10 F.

All tests were conducted at about 20 rpm for the first 500 cycles of stress. As a rule, the speed was then changed to 50 rpm until several thousand cycles were reached after which the highest speed between readings was about 150 rpm for the elevated temperature tests and various speeds up to 1000 rpm for the room temperature tests. In all cases the speed of reading was the same—20 rpm. Previous investigations (1, 6) have shown that damping and dynamic modulus of elasticity are frequency sensitive at room temperature. This is also true at elevated temperatures. However, at least at room temperature, the frequency of stress history does not affect the actual damping or modulus values that will be obtained after a given number of stress cycles.

Other details on testing equipment and procedures are described in previous publications (1, 4).

As indicated in the publications just referred to, the damping energy and dynamic modulus may be determined from the following relationships:

$$D = K\,(SH) \tag{3}$$

and
$$E_d = C\,(S/V) \tag{4}$$

where:

D = damping energy absorbed by the specimen, in-lb per cu in. per cycle of stress,

E_d = dynamic modulus of elasticity (secant value) during reversed-bending stress, psi,

S = amplitude of reversed stress in specimen, psi,

V = "vertical" (gravitational) deflection of the target T (toward table V in Fig. 1) due to bending stress in the specimen, in.,

H = horizontal traversal of the target T or its total lateral displacement (perpendicular to the direction of its gravitational deflection) caused by reversing the direction of rotation of the spindle, in., and

K and C = constants dependent only on specimen shape, weight, and center of gravity of the loading arm assembly, and the location of the target along the axis of the loading arm.

The method for evaluating the K constant in Eq 3 was the same as that discussed previously (1). For the tests reported in this paper, the initial room temperature static modulus of elasticity was determined for a given material by comparing the low stress deflection of the target under very slow rotation (less than 20 rpm) for specimens of that material with the corresponding deflections obtained with specimens of type 403 stainless steel having the same dimensions. The static modulus of elasticity value for type 403 was obtained from the results of a standard axial static tension test. The elevated temperature static modulus for a given material was determined by comparing the low stress deflections of the target under very slow rotation at test temperature with corresponding deflections measured at room temperature for the same specimen. Since for some of the powder metals studied in this program variations of about 5 per cent were observed in the static moduli of elasticity due to inhomogeneity of the material, the C constants in Eq 4 were determined as follows in order to unify the data and more readily reveal trends. Using Eq 6 in Reference (1), the dynamic modulus, E_d was calculated at a given stress history from the known values of E_s, D, and S. The corresponding vertical deflection, V, at this stress history was used in Eq 4 to determine C. This value of C was used thereafter at all stress histories to calculate the dynamic modulus from the vertical deflection values.

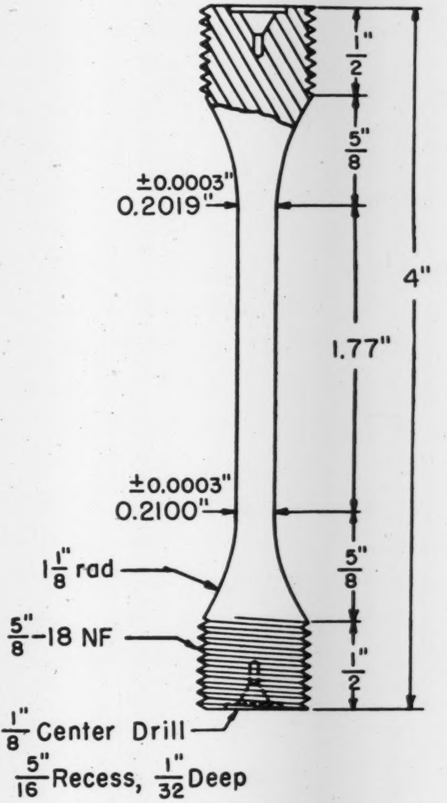

Fig. 3.—Type E Specimen for Damping, Elasticity, and Fatigue Testing.

TABLE I.—CHEMICAL COMPOSITION, PRODUCTION, AND TREATMENT.

Alloy	Chemical Composition, per cent																Production and Heat Treatment
	Carbon	Manganese	Phosphorus	Sulfur	Silicon	Nickel	Chromium	Molybdenum	Tungsten	Cobalt	Columbium	Iron	Aluminum	Titanium	Copper	Nitrogen	
S-816 (Material D)	0.40	0.70	0.014	0.016	0.51	20.68	19.79	3.46	4.46	43.40	3.80	2.50	...	...	...	...	Wrought bar stock 1⅝ in. diameter. Solution treatment—2150 F for 1 hr—water quenched. Aging treatment—1400 F for 16 hr—still air cooled.
Inconel X (Material E)	0.40	0.51	...	0.007	0.39	73.16	14.61	...	...	...	1.04	6.87	0.88	2.44	0.03	...	Wrought bar stock 1¾ in. diameter. Solution treatment—2000 F for 2 hr—fan air cooled. First aging—1550 F for 24 hr—still air cooled. Second aging—1300 F for 20 hr—still air cooled.
Type 403 (Material F)	0.12	0.37	0.016	0.022	0.26	0.44	12.67	...	...	...	...	86.1 (bal)	...	...	...	...	Wrought bar stock 1¾ in. diameter. Hardening treatment—1700 F for 15 min—fan air cooled. Tempering treatment—970 to 1020 F for 2 hr—air cooled. (Drawn to 30 to 36 Rockwell C)
Low-Carbon N-155 (Material NA)	0.15	1.74	...	...	0.37	19.4	21.7	2.76	1.90	19.0	0.76	32.1 (bal)	...	...	...	0.14	15.5 in. ingot hammer cogged to 2-in. squares. Rolled to 1-in. rounds at 2050 to 1830 F. Water quenched from 2200 F. Aging treatment—1400 F for 16 hr—air cooled.
TP-2-R (Material G)	...	...	...	...	...	...	...	100	...	...	...	...	...	...	...	...	Pure molybdenum powder containing microscopic traces of impurities, cold pressed and sintered in hydrogen bell, swaged to bar stock ⅞-in. diameter, forged to ⅝ in. flat between parallel dies. Annealing treatment—1750 F for 4 hr—hydrogen atmosphere.
TP-2-B (Material G_a)	...	...	...	...	...	...	...	98	2	...	...	...	...	...	...	...	Molybdenum with 2 per cent tungsten arc-cast (6-in. diameter). Hammered to 2⅝-in. diameter billets, rolled to ⅞-in. diameter, forged to ⅝-in. flat between parallel dies. Annealing treatment—1750 F for 4 hr—hydrogen atmosphere.
TP-1-2 (Material H)	...	...	...	...	...	...	...	...	...	...	...	85[a]	...	...	15[a]	...	High purity iron powder (approximately 98.5 per cent pure) cold pressed, sintered, coined, and "infiltrated" with a molten copper alloy to obtain 100 per cent density. Iron contained following approximate impurities before sintering: C............ 0.03 per cent P............ 0.006 per cent Mn.......... 0.4 per cent Oxides....... 1.0 per cent Si............ 0.1 per cent TP-1 material given standard solutions and aging treatment to obtain maximum tensile strength.
TP-1-3 (Material H_a)	...	...	...	...	...	...	...	...	...	...	...	85[a]	...	...	15[a]	...	Same as TP-1-2 material but giving modified solution and aging treatment to obtain high ductility.

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[a] Approximate.

DESIGN AND PREPARATION OF TEST SPECIMEN

The test specimen utilized in this work is shown in Fig. 3. The tapered test section is so designed as to produce equal maximum bending stress along the length of the test section under the cantilever loading. In the interest of uniformity of stress, it is also desirable to use hollow, thin-walled specimens (1). However, specimen preparation difficulties for several of temperature-resistant materials necessitate use of the small, solid specimens shown. Since in these solid specimens all stresses from zero to maximum are always present, the damping energy determined is an average value for stresses between zero and maximum.

Final polishing of the specimens was done in a special abrasive belt machine (8).

TEST MATERIALS AND PROGRAM

The test materials used in this program and the testing temperatures for each are given below:

S-816	Room temperature, 900 F and 1600 F
TP-2-R	Room temperature and 900 F
TP-2-B	Room temperature and 900 F
Type 403	Room temperature and 500 F
TP-1-2	Room temperature and 500 F
Inconel X	Room temperature
Low-carbon N-155	1500 F

The above elevated temperatures were selected for the tests because 500 F is close to the average temperature of the blades in the compressor section of a jet engine, 900 F is about the highest temperature that can be used with the TP-2 materials without using a protective atmosphere, and 1600 F is the approximate operating temperature of the blades in the turbine section of a jet engine. There is considerable creep data available for N-155 at 1500 F; hence, the selection of that test temperature.

Chemical compositions, heat treatment data, and static physical properties are given in Tables I and II.

The aims of this investigation were to secure data of engineering value on the dynamic properties of damping, elasticity, and fatigue; to present them in a clear fashion showing the dependence of these properties on certain other variables; and lastly to present methods for the comparison and evaluation of these dynamic properties of materials. An attempt to explain the reasons for the different behaviors obtained and a metallurgical investigation of the types of fracture encountered or the changes taking place in the structure of the materials during the course of the tests are extremely important phases of the entire study that are unfortunately beyond the scope of this present paper.

TABLE II.—STATIC PHYSICAL PROPERTIES.

Material	Temperature, deg Fahr	Yield Strength, 0.2. per cent offset, psi	Tensile Strength, psi	Elongation, per cent	Reduction in Area, per cent	Modulus of Elasticity, psi
S-816	RT	67 000	140 000	35	29	34.0×10^6
	900	55 000	125 000	20	10	29.4
	1600	39 600	59 000	25	30.2	24.0
TP-2-R	RT		75 500	(0	0	45
	900	64 500	66 200	12	48	42.7
TP-2-B	RT	104 500	120 000	4.5	4.8	48
	900	67 000	71 000	19	74.5	44.4
Type 403	RT	138 000	152 000	17.5	64	29.0
	500	122 000	139 000	15	60	26.5
TP-1-2	RT	107 000	121 000	2.0	3.0	24.5
	500	89 000	99 500	2.0	4.3	23.0
TP-1-3	RT	65 000	77 000	9.7		24.5
	500		67 500	12.5		23.0
Inconel X	RT	92 000	162 000	24	30	31
Low-carbon N-155	1500	35 800	44 600	23	27	

TEST DATA

Records of the damping and elasticity data kept during the course of constant stress rotating-bending fatigue tests of a given material afford information from which basic diagrams of the type shown in Figs. 4 and 7 are constructed. Figure 4 is a plot of the original data of damping energy *versus* the number of reversed stress cycles for the tests on S-816 alloy. Each curve represents the data from a single fatigue test of a given virgin specimen (one not previously subjected to stress) brought either to failure in above fatigue strength tests or to several million cycles in below fatigue strength tests. Thus, each curve shows the effect of number of cycles, N, in the stress history on the damping energy, D, during a test at constant reversed bending stress, S. By testing other specimens, each at a different stress, the dependence of damping on stress magnitude is obtained. The series of curves represents a so-called *D-N-S* diagram for the material at that temperature. By carrying out such a program at different temperature levels, the effect of temperature is determined.

In a manner similar to the above, Fig. 7 presents the dynamic modulus of elasticity data for S-816, showing by means of an E_d-N-S diagram, the effect of stress history and stress magnitude on the dynamic modulus.

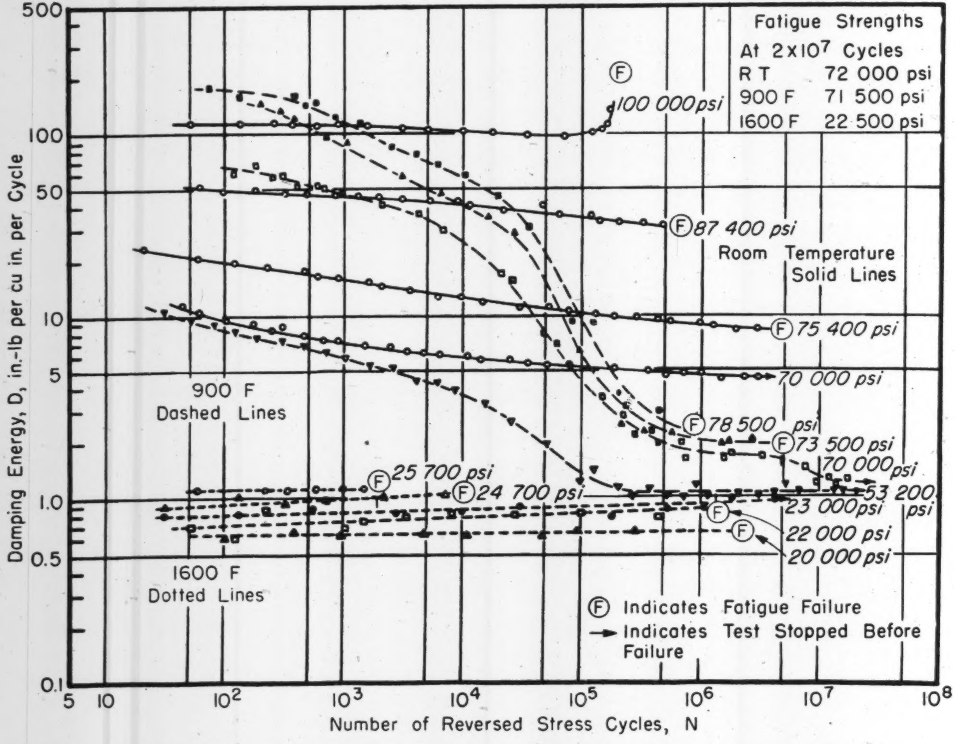

FIG. 4.—*D-N-S* Diagram Showing the Effects of Several Magnitudes of Sustained Reversed Stress on the Relationship Between Damping Energy and Number of Cycles for S-816 Alloy (Material D) at Room Temperature, 900 F, and 1600 F.

Figure 22 presents usual *S-N* fatigue curves for the results of room temperature and elevated temperature tests of all materials.

The other diagrams presented in this paper were prepared by replotting and combining in different ways the original damping, elasticity, and fatigue data secured at the various temperatures. These new diagrams are intended to increase understandability and usability in design and in material comparison studies.

It should be emphasized at this point that the entire testing program was designed to be exploratory in nature. Only a few specimens of each material were tested at a given temperature to obtain the damping, elasticity, and fatigue data. It is felt that since so little is known regarding damping and related dynamic properties, exploratory tests on several materials are more revealing than an equal effort spent on an intensive study of one of these materials. That this extensive rather than intensive approach was desirable is indicated by the wide variety of behavior patterns displayed by the various materials.

It should be pointed out also that the fatigue, damping, and elasticity diagrams presented involve, in some cases, extrapolating and smoothing out of the original data obtained. Therefore, the values and patterns indicated should, in some cases, be considered qualitative and comparative studies rather than precise quantitative data.

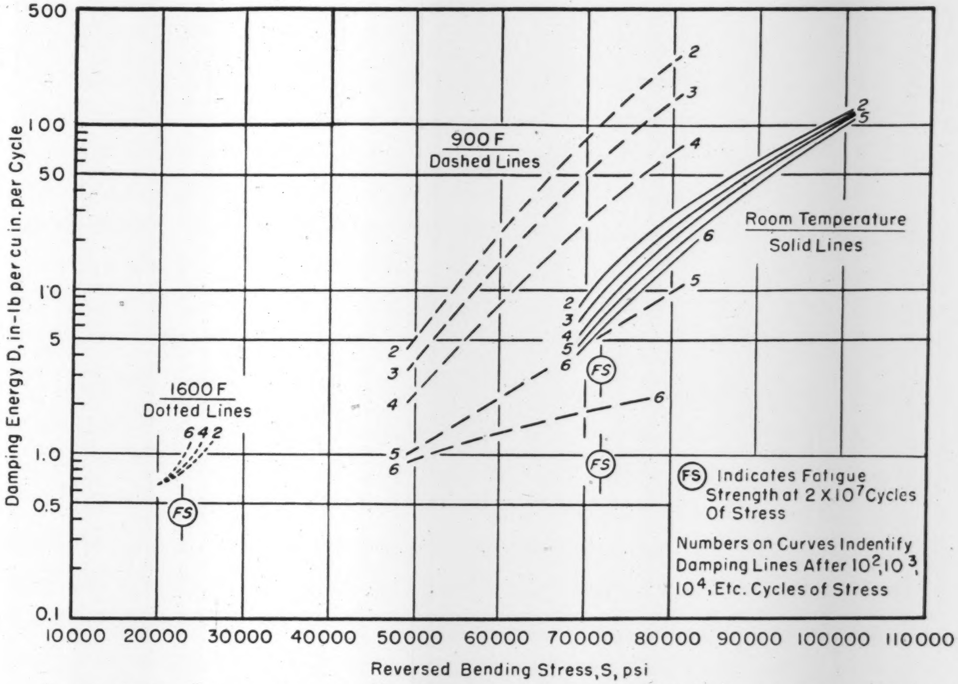

FIG. 5.—*D-S-N* Diagram Showing the Effects of Several Numbers of Sustained Reversed Stress Cycles on the Relationship Between Damping Energy and Stress Magnitude for S-816 Alloy (Material D) at Room Temperature, 900 F, and 1600 F.

DISCUSSION OF DATA

Damping, Elasticity, and Fatigue Properties:

Figure 4 is presented as a typical *D-N-S* diagram in order to show a complete set of original damping data. The plot is of particular interest because this material, S-816, was tested at three different temperatures and also because the curves (at 900 F) display greater changes in damping energy with stress history than do those for any of the other materials tested. The room temperature data shown in solid lines indicate a definite decrease in damping with increasing number of cycles until fracture occurred for all specimens except the one tested at 100,000 psi. This curve displays a sharp upward trend near the end which may be significant since it occurred during a period representing more than half of the life of the specimen (cycles, N, plotted logarithmically).

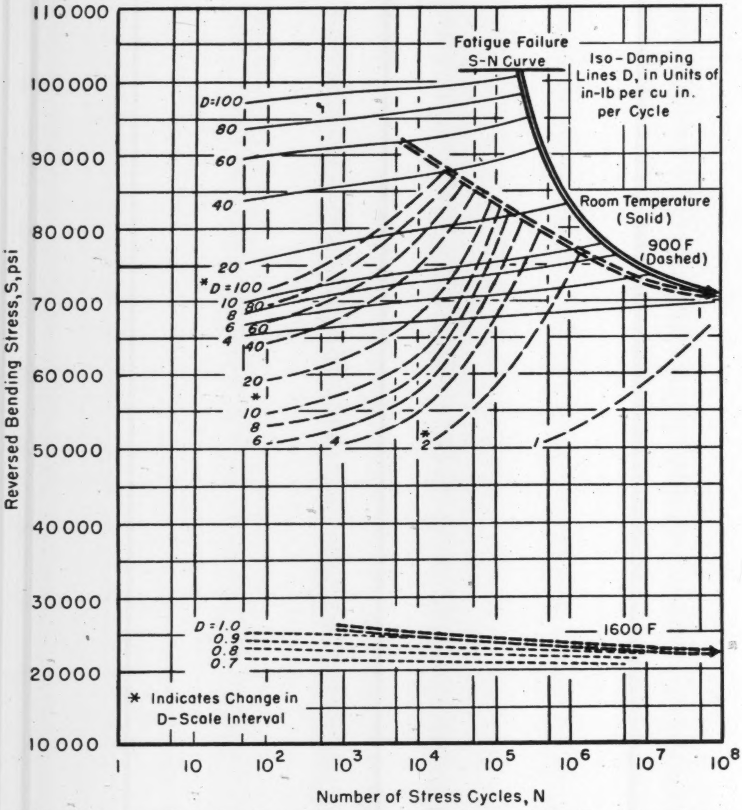

FIG. 6.—*S-N-D* Diagram Relating Stress, Number of Cycles, and Iso-Damping Contour Lines for S-816 Alloy (Material D) at Room Temperature, 900 F, and 1600 F.

For the tests at 900 F, curves shown as dashed lines, there is an even more pronounced decrease in damping with stress history than at room temperature. The decrease is particularly rapid in the region of 10^4 to 10^6 cycles of stress. For the specimen tested at a stress of 78,500 psi, which is about 10 per cent above the fatigue strength, the damping decreased from a value of 200 in-lb per cu in. per cycle at 50 cycles to about 2 in-lb per cu in. per cycle at 10^6 cycles of stress, representing a decrease to 1 per cent of its 50 cycle value. At the lower stresses the change is somewhat smaller than this. So that time at temperature (900 F) is not thought to be the sole cause of this decrease, it should be noted that the aging treatment for this material consisted of 16 hr at 1400 F followed by still air cooling. Furthermore, when the specimen tested at 53,200 psi at 900 F (which did not break in more than 10 million cycles) was retested at a higher stress, a curve similar in shape to those for the virgin specimen was obtained. The rapid decrease in damping again occurred at about the same number of cycles.

The damping data for 1600 F shown as dotted lines in Fig. 4 display little change with stress history exhibiting only a slight *increase* as the number of cycles becomes larger. The specimen at 20,000 psi which is below the reported fatigue strength at 1600 F was overheated before testing and it is thought that this adversely affected its life.

It should be pointed out that the damping values do not always decrease with an increase in number of cycles at room temperature. Many different patterns of behavior have been observed; for example, the damping for TP-2-B increases continuously with an increase in the number of cycles; type 403 displays an initial decrease followed by an increase to a peak after which the values decrease if the specimen does not previously fracture. The data for TP-1-2 show damping values decreasing to a minimum followed by a steady increase to fracture; material TP-1–3 displays practically no change in damping with number of cycles; and Inconel X has very high initial damping followed by a period of practically no change after which the damping increases sharply till failure. The general trends for a given material may be the same at elevated temperature as at room temperature, as in the case of TP-2-B for room temperature and 500 F, or they may be reversed as in the case of S-816 at room temperature and 1600 F. Qualitative indications of these trends are discussed later in connection with the *S-N-D* diagrams presented.

The data for S-816 diagrammed in Fig. 4 are replotted in Fig. 5. This diagram shows damping, *D*, at three temperatures as a function of the magnitude of reversed bending stress, *S*, with lines marked 2, 3, 4, 5, and 6 connecting the damping values after 10^2, 10^3, 10^4, 10^5, and 10^6 cycles, *N*, of stress. This *D-S-N* diagram affords a convenient method for comparing the damping behavior of S-816 at the three temperatures. The flags enclosing the letters F.S. indicate the fatigue strengths of the material. It is interesting to compare the damping values at different temperatures for stresses corresponding to the fatigue strengths. The room temperature damping after 100 cycles at a stress equal to the fatigue strength is about one-fifth that of the damping at 900 F at a stress equal to the 900 F fatigue strength and about twenty times the corresponding value for 1600 F. After 10^6 cycles of stress, the room temperature value is about three times the corresponding 900 F value and about six times that at 1600 F.

A method of showing the fatigue properties along with the damping properties for a given material in one diagram is shown in Fig. 6. This presents an *S-N-D* diagram for S-816 alloy showing the data for the three test temperatures. The plot is easily made from the *D-S-N* diagram of Fig. 5. It shows the relationships between the three variables by the use of iso-damping contour lines *D* on a plot of stress magnitude, *S*, *versus* the number of stress cycles, *N*. In Fig. 6 the data for room temperature are shown in solid lines, those for 900 F in dashed lines, and those for 1600 F in dotted lines. The double curve in each case represents the fatigue failure *S-N* curve.

In interpreting an *S-N-D* diagram, the rules relating to contour lines of elevation on a topographic map are useful. For example, in proceeding toward the right in Fig. 6 along a constant stress line, the crossing of iso-damping lines of successively lower values for both room temperature and 900 F data indicates a downhill trend in damping energy. Conversely, with the data for 1600 F, an uphill trend is seen in damping energy at constant stress as the number of stress cycles is increased. These behaviors are, of course, merely another method for showing the trends indicated in the plot of Fig. 4.

Of additional interest in Fig. 6 is the observation that the fatigue strength for 20 million cycles at 900 F is practically the same as the room temperature value, but the 1600 F fatigue strength is only about one-third of the room temperature value.

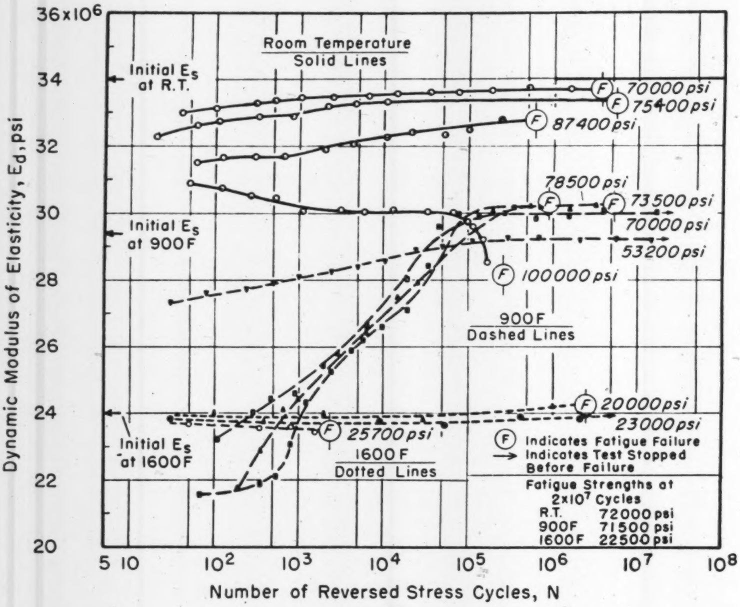

FIG. 7.—E_d-*N*-*S* Diagram Showing the Effects of Several Magnitudes of Sustained Reversed Stress on the Relationship Between Dynamic Modulus of Elasticity and Number of Cycles for S-816 Alloy (Material D) at Room Temperature, 900 F, and 1600 F.

As pointed out in a previous paper (1), the variation of dynamic modulus of elasticity with number of cycles of stress is, in general, reciprocal to the change in damping capacity. Figure 7 is an E_d-*S*-*N* diagram for S-816 alloy at the three testing temperatures. Comparing this plot with the corresponding *D*-*N*-*S* diagram of Fig. 4, the general reciprocal relationship mentioned above is apparent. For the specimens tested at room temperature, an increase in dynamic modulus is observed at all stresses except the highest —100,000 psi. This curve shows a decreasing trend to fracture where the value recorded is approximately 80 per cent of the initial modulus value. The data for all of the specimens at 900 F show increasing values of E_d with an increase in number of cycles. The values for a small number of cycles are approximately 70 per cent of the initial static modulus value at 900 F, while the highest indicated are some 3 per cent above this figure. Changes in the static modulus of elasticity after exposure to reversed stress at elevated temperature for a large number of cycles have been determined for S-816. For one specimen at 1600 F, a stress history of one million cycles at 1600 F at a stress slightly below the fatigue strength resulted in an increase of slightly more than 1 per cent over the value for the virgin specimen. (For one specimen of N-155 tested at 1500 F at a stress slightly above the fatigue strength, an increase of about 3 per cent in the static modulus was measured after 100,000 cycles of stress.) The fatigue strengths of S-816 at the three temperatures are indicated in the corner of Fig. 7 to provide a measure of the proximity of the test stress to the fatigue strength in each case. The static moduli for the virgin specimens at each temperature are indicated on the left side so that comparisons with the values for dynamic modulus may be made.

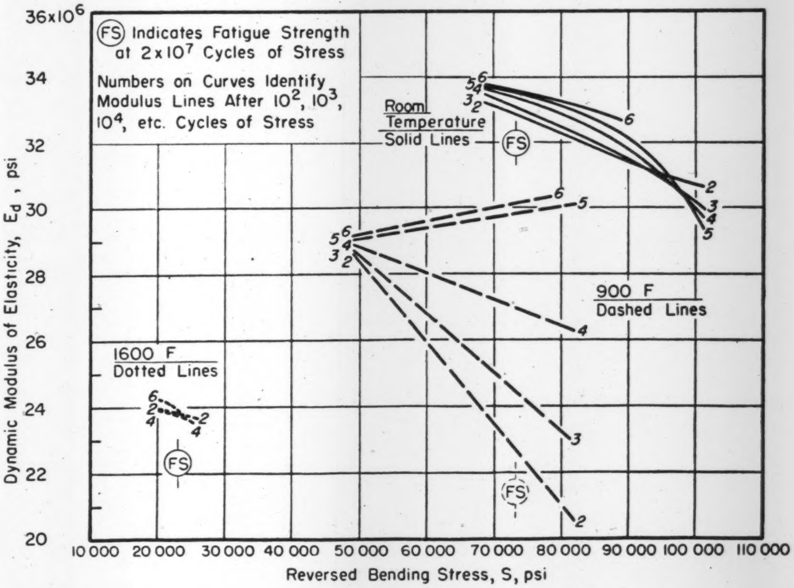

FIG. 8.—E_d-S-N Diagram Showing the Effect of Several Numbers of Sustained Reversed Stress Cycles on the Relationship Between Dynamic Modulus of Elasticity and Stress Magnitude for S-816 Alloy (Material D) at Room Temperature, 900 F, and 1600 F.

The dynamic modulus does not for all materials increase with an increase in the number of stress cycles. There are just as many different trends and patterns in the behavior of this property as in the case of damping energy. However, the reciprocal relationship to the damping values does generally exist.

The data for the dynamic modulus of S-816 in Fig. 7 are replotted in Fig. 8 as an E_d-S-N diagram to facilitate explanation. The diagram shows dynamic modulus E_d for the three temperatures as a function of reversed bending stress, S, with lines marked 2, 3, 4, 5, and 6 connecting the dynamic modulus values after 10^2, 10^3, 10^4, 10^5, and 10^6 cycles of stress. It shows clearly the large changes in elasticity at 900 F with both stress magnitude and stress history when compared with the smaller changes at the other two temperatures. The F.S. flags are again used to indicate the fatigue strengths at the different temperatures.

The summary diagram for the elasticity and fatigue properties for S-816 alloy at the three test temperatures (corresponding to Fig. 6 for the damping and fatigue properties) is the S-N-E_d diagram shown in Fig. 9. This plot shows the relationships between three variables by showing iso-modulus of elasticity contour lines E_d on a plot of stress magnitude S *versus* the number of stress cycles N. Again solid, dashed, and dotted lines indicate the data for room temperature, 900 F, and 1600 F. The static moduli of elasticity for the virgin specimens at each temperature are presented in the corner of the diagram for comparison purposes. Such diagrams are shown in Figs. 10 and 11 for TP-2-R (pure molybdenum), in Figs. 12 and 13 for TP-2-B (molybdenum plus 2 per cent tungsten), in Figs. 14 and 15 for type 403, and in Figs. 16 through 19 for the TP-1 (copper infiltrated sintered iron powder). Data are presented for each of these materials investigated at two temperature levels, room temperature and 900 F for three of the materials, and room temperature and 500 F for three others.

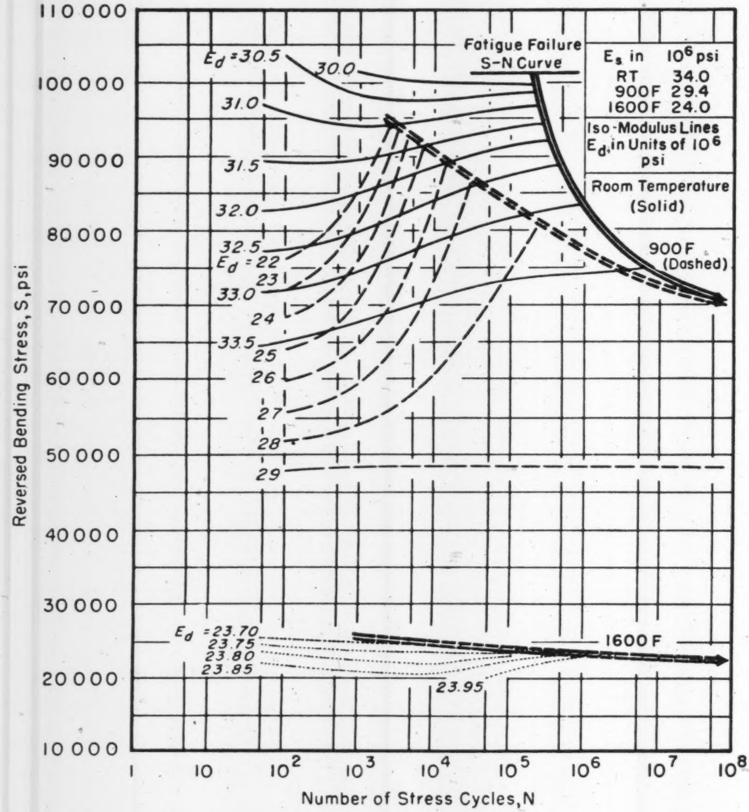

FIG. 9.—S-N-E_d Diagram Relating Stress, Number of Cycles, and Iso-Modulus of Elasticity Contour Lines for S-816 Alloy (Material D) at Room Temperature, 900 F, and 1600 F.

The general trends of the iso-modulus lines are seen to be similar to those of the iso-damping lines in Fig. 6, although the change in elasticity is reciprocal to the change in damping.

Companion S-N-D and S-N-E_d diagrams present the damping, elasticity, and fatigue data for a given material.

The two TP-2 materials display generally similar trends for the iso-damping and iso-modulus lines. Sloping downward to the right, the iso-damping lines indi-

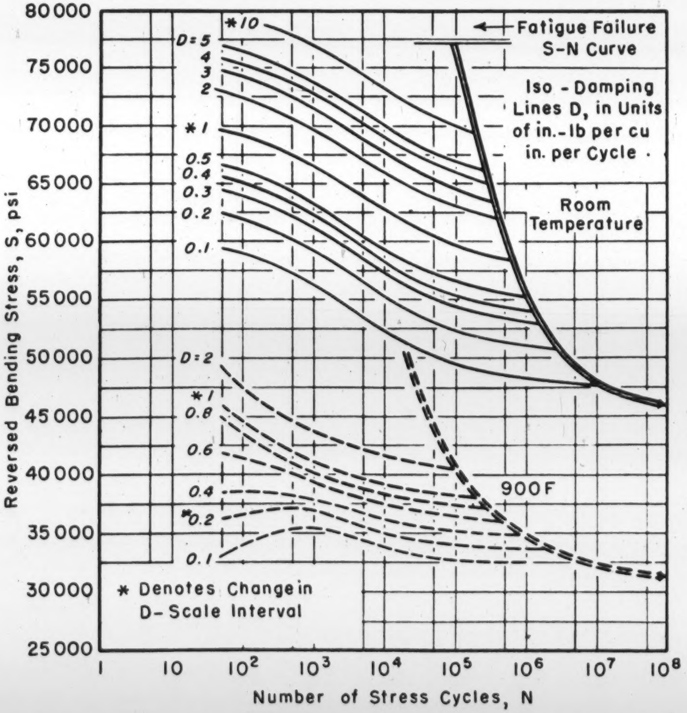

FIG. 10.—*S-N-D* Diagram Relating Stress, Number of Cycles, and Iso-Damping Contour Lines for TP-2-R (Material G) at Room Temperature and 900 F.

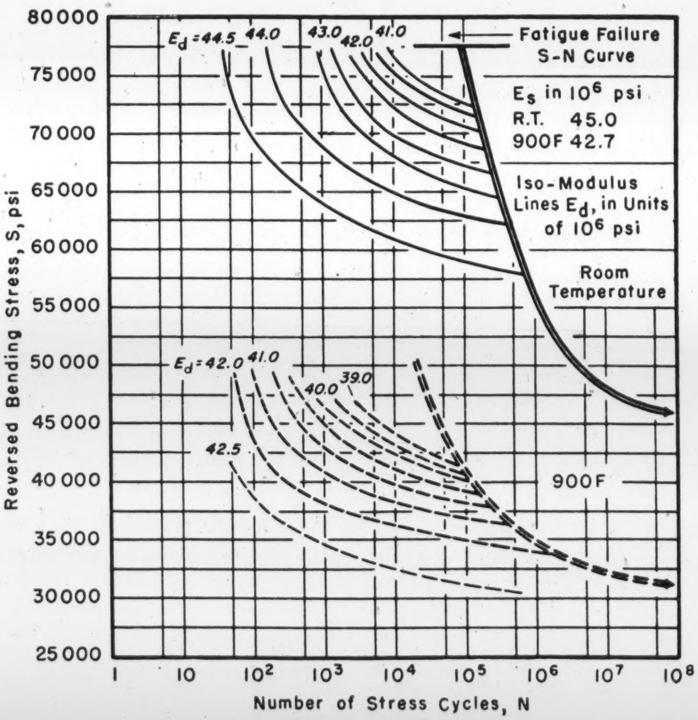

FIG. 11.—*S-N-E_d* Diagram Relating Stress, Number of Cycles, and Iso-Modulus of Elasticity Contour Lines for TP-2-R (Material G) at Room Temperature and 900 F.

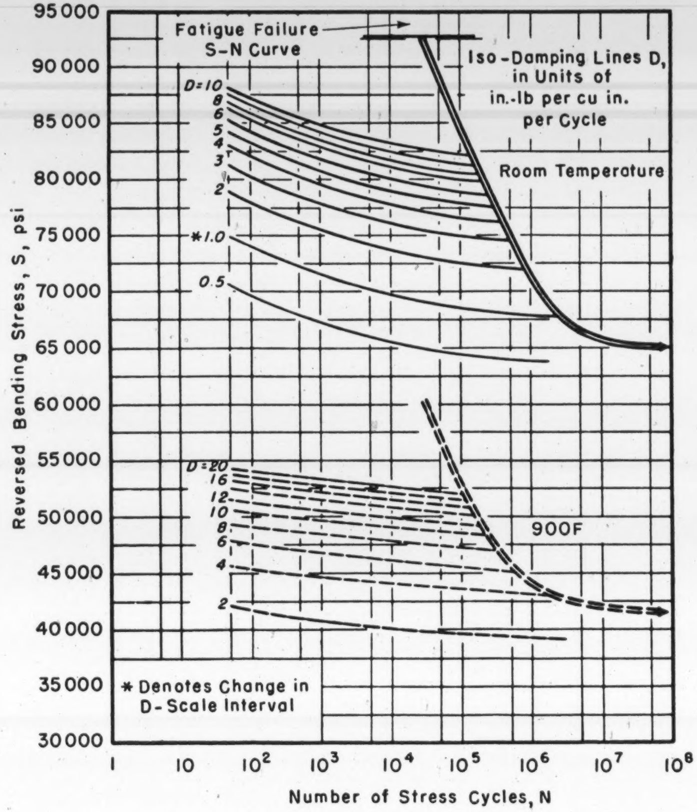

FIG. 12.—*S-N-D* Diagram Relating Stress, Number of Cycles, and Iso-Damping Contour Lines for TP-2-B (Material G_a) at Room Temperature and 900 F.

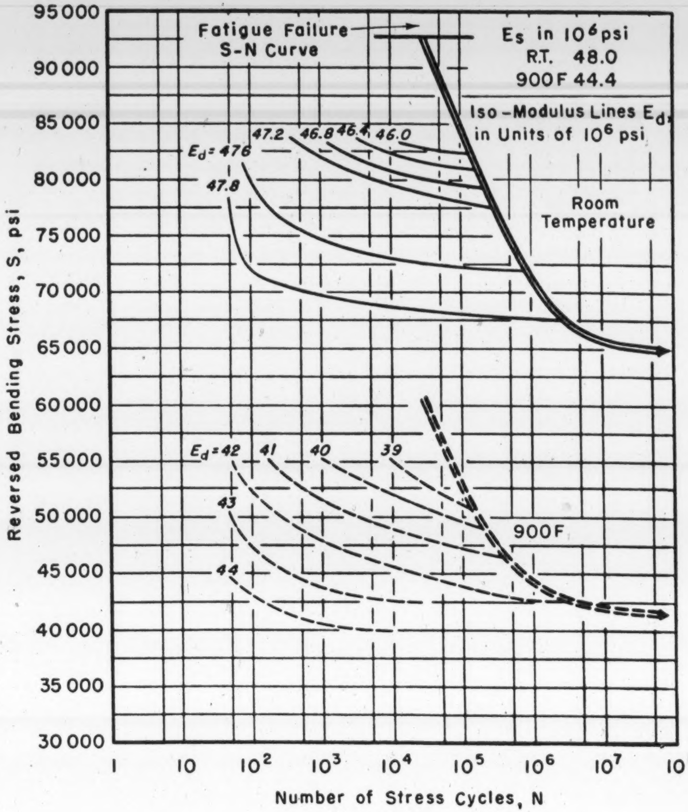

FIG. 13.—*S-N-E_d* Diagram Relating Stress, Number of Cycles, and Iso-Modulus of Elasticity Contour Lines for TP-2-B (Material G_a) at Room Temperature and 900 F.

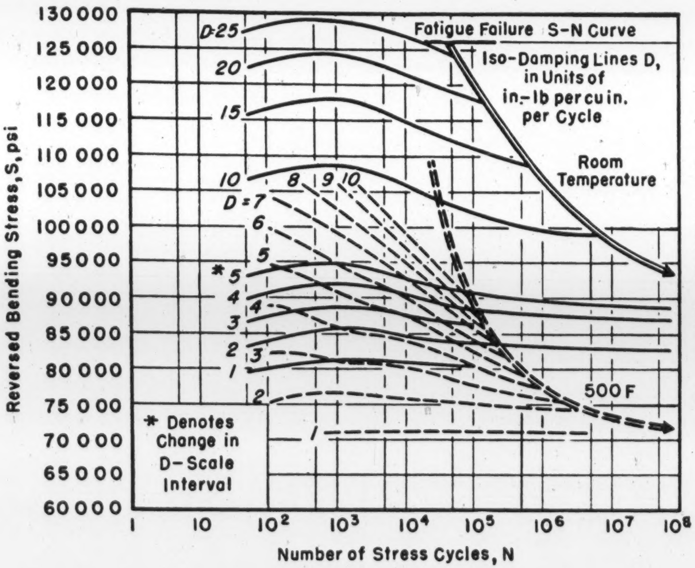

FIG. 14.—*S-N-D* Diagram Relating Stress, Number of Cycles, and Iso-Damping Contour Lines for Type 403 Alloy (Material F) at Room Temperature and 500 F.

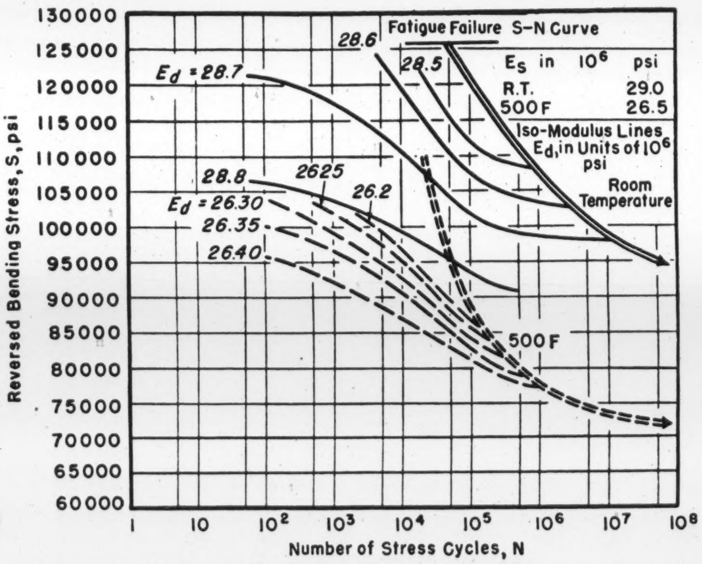

FIG. 15.—*S-N-E*$_d$ Diagram Relating Stress, Number of Cycles, and Iso-Modulus of Elasticity Contour Lines for Type 403 Alloy (Material F) at Room Temperature and 500 F.

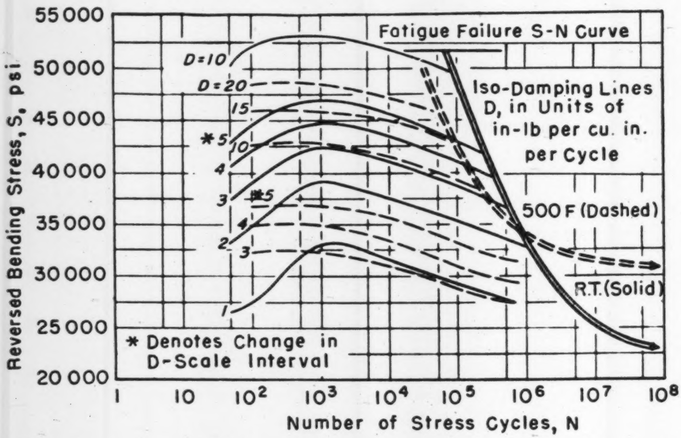

FIG. 16.—*S-N-D* Diagram Relating Stress, Number of Cycles, and Iso-Damping Contour Lines for TP-1-2 (Material H) at Room Temperature and 500 F.

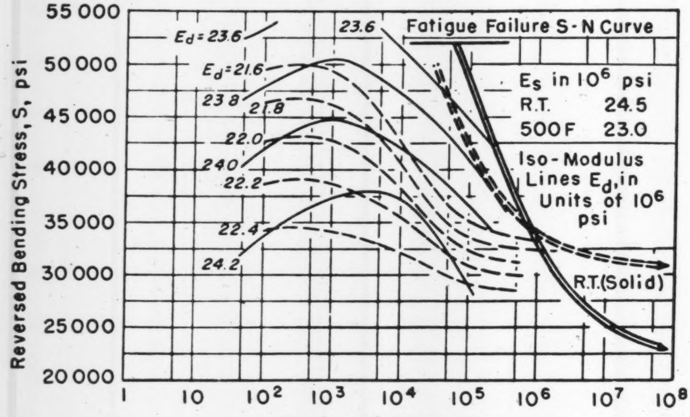

FIG. 17.—*S-N-E_d* Diagram Relating Stress, Number of Cycles, and Iso-Modulus of Elasticity Contour Lines for TP-1-2 (Material H) at Room Temperature and 500 F.

cate an uphill trend in damping values during a constant stress test since a constant stress line intersects lines of increasing *D* values as the number of cycles *N* increases. The E_d lines also sloping downward to the right indicate a downhill trend in modulus values since a similar stress line intersects iso-modulus lines of lower values with increasing number of stress cycles *N*. The patterns displayed by type 403 and TP-1-2 are generally similar at both room temperature and 500 F, although the stress levels for corresponding damping values at a given number of cycles are much higher for type 403. The data for TP-1-3 display the least change in damping and elasticity values with number of cycles of any of the materials tested (Figs. 18 and 19). A noteworthy point in connection with the data for the TP-1 materials is that in both cases the fatigue failure curves at 500 F cross over the room temperature curves after about one million cycles so that the fatigue strengths at 20 million cycles for 500 F are higher than the room temperature values. Other characteristics of the diagrams might be described but it is felt that examination of the graphs will make the salient points obvious.

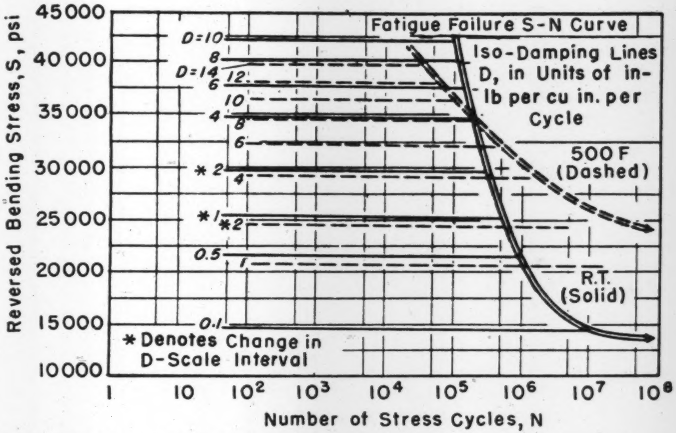

FIG. 18.—*S-N-D* Diagram Relating Stress, Number of Cycles, and Iso-Damping Contour Lines for TP-1-3 (Material H_a) at Room Temperature and 500 F.

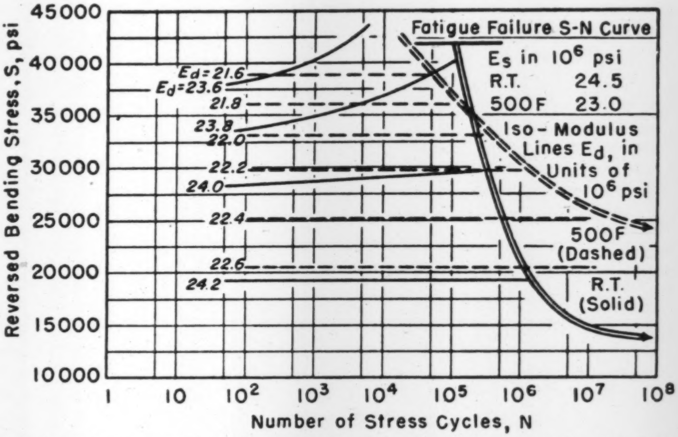

FIG. 19.—*S-N-E_d* Diagram Relating Stress, Number of Cycles, and Iso-Modulus of Elasticity Lines for TP-1-3 (Material H_a) at Room Temperature and 500 F.

Limited data on Inconel X were obtained by testing type Y specimens (solid cylindrical bars, 0.43 in. in diameter by $3\frac{1}{2}$-in. test length, tapered for constant stress). The S-N-D diagram for this material at room temperature is presented in Fig. 20. The dome shaped iso-damping lines indicate a sharp initial decrease in damping followed by a period of no change and then by sharply increasing damping values in the tests run at constant stress. There were no specimens of this material tested at stresses near the long time fatigue strength so the data are all for comparatively short time tests of below 10^5 cycles. The elasticity data for this material are not presented.

Limited data were also procured on the dynamic properties of low carbon N-155 alloy (material NA) at 1500 F. An *S-N-D* diagram is presented in Fig. 21 for this material. The iso-damping lines indicate relatively little change in damping properties during the life of a specimen tested at constant stress.

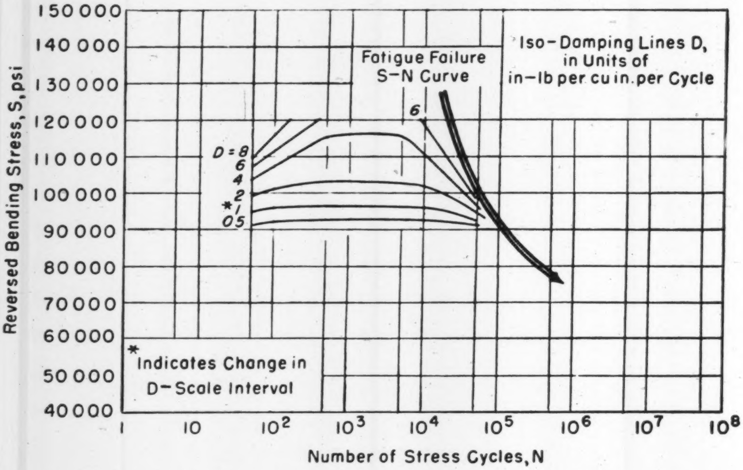

FIG. 20.—*S-N-D* Diagram Relating Stress, Number of Cycles, and Iso-Damping Contour Lines for Inconel X (Material E) at Room Temperature Obtained Using Type Y Specimens (Test Length $3\frac{1}{2}$ in., Average Diameter 0.428 in).

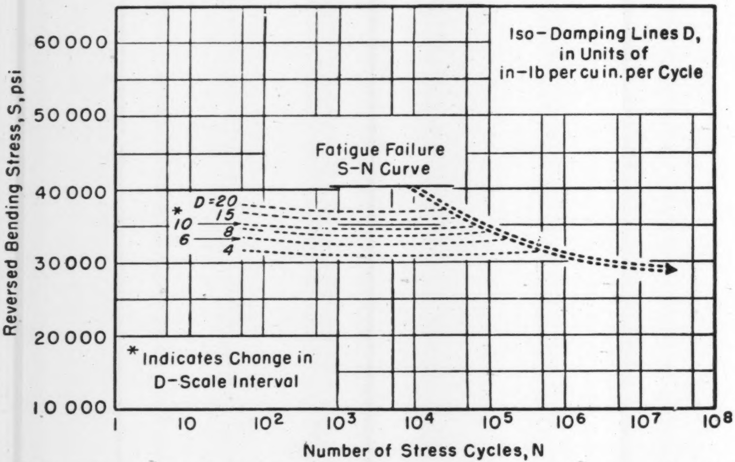

FIG. 21.—*S-N-D* Diagram Relating Stress, Number of Cycles, and Iso-Damping Contour Lines for Low-Carbon N-155 Alloy (Material NA) at 1500 F.

The actual fatigue *S-N* data which were used in plotting the fatigue curves shown in Figs. 8 through 21 are presented in Fig. 22. The results of the room temperature tests are shown in the first of the two diagrams. The fine lines indicate extrapolated portions of the curves. Although the scatter in the fatigue data is generally relatively small, these data must be considered only approximate since so few specimens were used for each curve. The elevated temperature fatigue data are presented for type 403, TP-1-2, and TP-1-3 at 500 F; for S-816, TP-2-R, and TP-2-B at 900 F; for N-155 at 1500 F; and for S-816 at 1600 F. In this figure, dashed lines are used for the 500 F data, dotted for 900 F, and dash-dot lines for temperatures above 900 F. One observation made concerning the *S-N* curves of Fig. 22 is that they generally become flatter as the temperature level is raised.

An interesting behavior was that noted for the specimens of S-816 fatigue tested at 1600 F. The specimens did not undergo brittle fracture at the end of a fatigue test, but their stiffness suddenly decreased to such an extent as to allow the loading arm to strike the microswitch and stop the test. Such behavior is not apparent from Fig. 7 since the action occurred so abruptly that no readings were taken immediately preceding the end of a test. This sudden decrease in stiffness did not occur with the same material at 900 F. At this temperature the normal fatigue type of fracture was observed. This was also the case with all of the other elevated temperature speci-

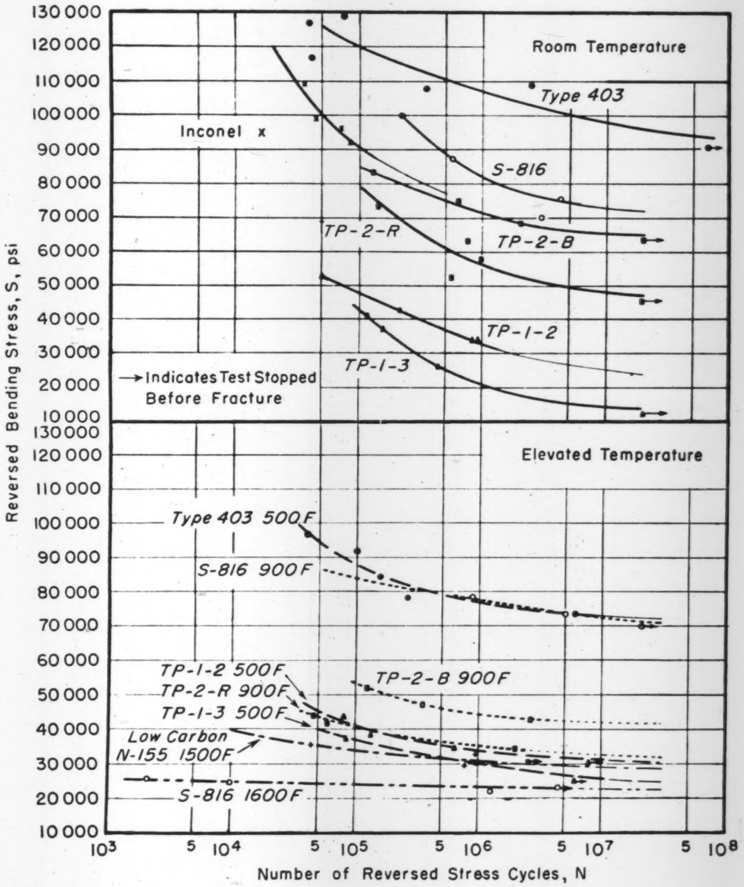

FIG. 22.—*S-N* Fatigue Curves for Various Temperature-Resistant Materials.

mens including those of low carbon N-155 at 1500 F.

A summary of the fatigue strength data obtained in these tests is presented in Table III.

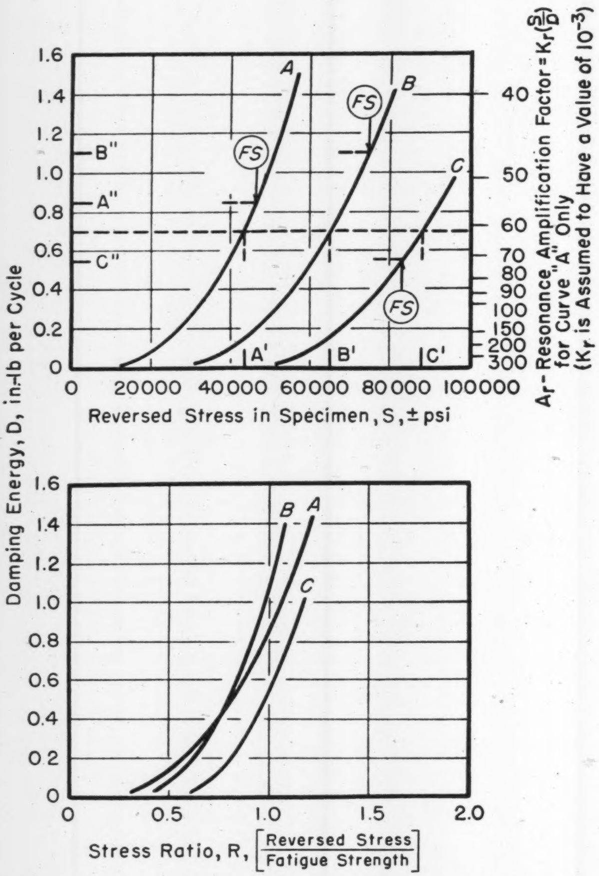

FIG. 23.—Hypothetical Curves for Three Materials Illustrating Methods of Comparing Damping Properties.

Comparison of the Damping and Fatigue Properties:

When making a choice among several materials for a given dynamic application, not only are the three properties of damping, dynamic modulus, and fatigue strength important individually but also their interrelationships are of considerable engineering significance. In comparing the damping properties of several materials, not only the magnitude of damping energy at a given stress is im-

TABLE III.—FATIGUE STRENGTHS IN PSI FOR 20 MILLION CYCLES OF REVERSED BENDING STRESS.

Material	Temperature, deg Fahr				
	Room	500	900	1500	1600
S-186	72 000	...	71 500	...	22 500
TP-2-R	47 000	...	32 000	...	...
TP-2-B	65 000	...	42 000	...	...
Type 403	96 000	72 000	...	...	...
TP-1-2	24 000	31 000	...	...	...
TP-1-3	14 000	25 000	...	...	...
Inconel X	57 000[a]	...	...	...	...
Low carbon N-155	...	...	...	29 000	...

[a] Approximate value supplied by Thompson Products Corp.

portant but also the proximity of that stress to the fatigue strengths of the various materials should be considered.

To clarify two methods for comparing the damping properties of a set of materials tested at a given temperature, hypothetical curves for three materials will be discussed. In the upper portion of Fig. 23 are shown curves for three materials A, B, and C which represent a plot to linear scales of damping energy D *versus* stress magnitude S for the three materials after a given number of stress cycles. The damping curves for actual materials do have these general shapes when plotted to linear scales. The flags marked F.S. indicate the fatigue strengths for the materials with C having the highest value and B and A following in that order. On the right side of the figure is indicated a scale for the resonance amplification factor for curve A only, assuming a reasonable value for K_r of 10^{-3}. This factor determines the alternating stress at resonance as given by Eq 2.

If the damping properties of these three materials are compared on the basis of *equal stress magnitude*, material A would be preferred to material B since it has the greater energy absorption capacity at all values of stress. Likewise, material B would be chosen in preference to material C. However, the relative fatigue strengths of the three materials should also be considered. When operating near resonance, a given machine part may be required to absorb a given amount of input vibrational energy if the near resonance stresses are to be kept within safe limits. Thus, in making a choice of materials for a part subject to near resonance service, comparison should be made on the basis of equal energy absorbed rather than on the basis of equal stress. The amplitude of vibration and, therefore, the stress would increase until the energy dissipated through damping was equal to the vibrational energy input. For example, with the three materials under consideration, suppose that the amount of energy required to be absorbed by the material is 0.7 in-lb per cycle. A horizontal line drawn at this value of damping energy in the upper diagram of Fig. 23 indicates the three materials would be operating at stresses shown by values A′, B′, and C′ respectively on the abscissa. In comparing these stresses with the corresponding fatigue strengths, material A operates slightly below its fatigue strength, material B considerably below, and material C would be significantly above its fatigue strength.

In order to clarify further the relationship between damping and fatigue behaviors, the damping data for the three materials A, B, and C are replotted as shown in the lower portion of Fig. 23. In this graph, the abscissa is the ratio of the reversed stress to the fatigue strength of the material. At stresses corresponding to their fatigue strengths, materials A, B, and C have damping energy values as given by A″, B″, and C″ in the upper portion of Fig. 23. These values are plotted in the lower diagram at a stress ratio of 1.0. In comparing the relative damping properties of the three materials in this manner, material C is the least effective energy absorber. Material A has the highest damping at low stress ratios, but material B exhibits superior properties to A for values of stress ratio in excess of 0.75.

The foregoing discussion is not meant to imply that a material of high fatigue strength is always inferior with respect to damping when compared on the basis of stress ratio with materials of lower fatigue strengths (see Reference (3) for example). On the contrary, in many cases materials of high fatigue strength often exhibit higher damping capacity at stress

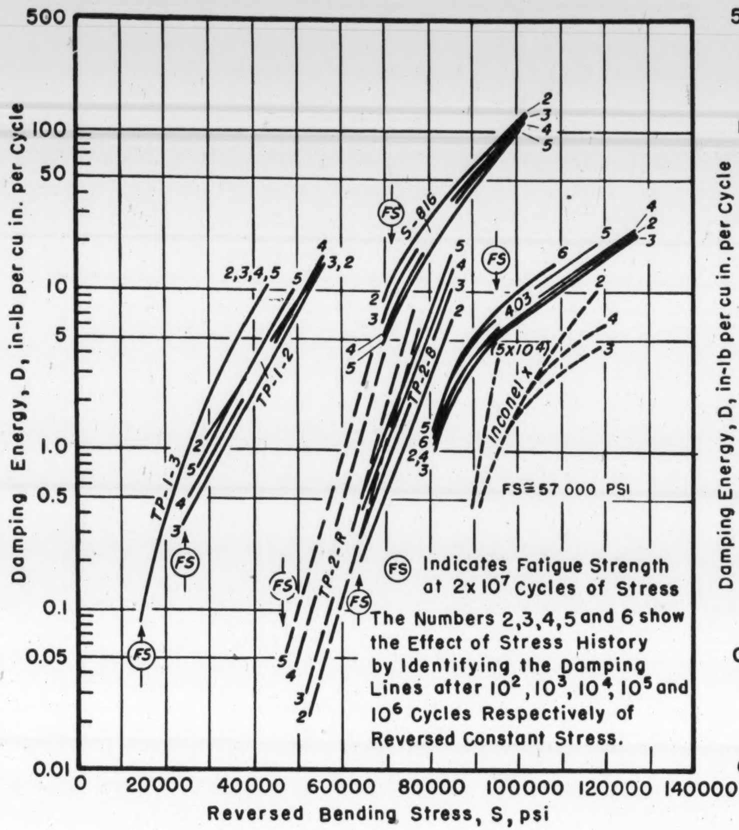

FIG. 24.—*D-S-N* Diagrams for Several Temperature-Resistant Materials Comparing Damping Energy at Room Temperature as a Function of Stress History and Stress Magnitude.

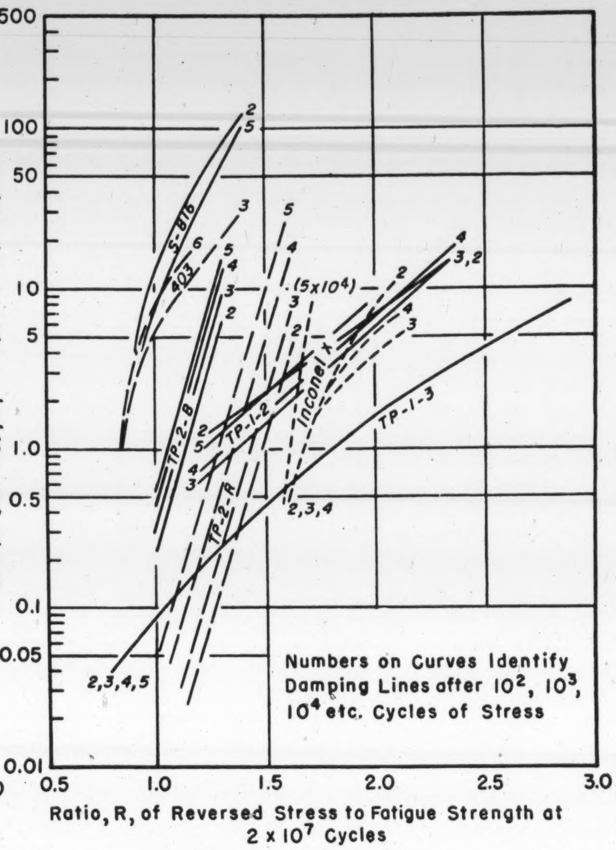

FIG. 25.—*D-R-N* Diagrams for Several Temperature-Resistant Materials Comparing Damping Energy at Room Temperature as a Function of Stress History and Stress Ratio.

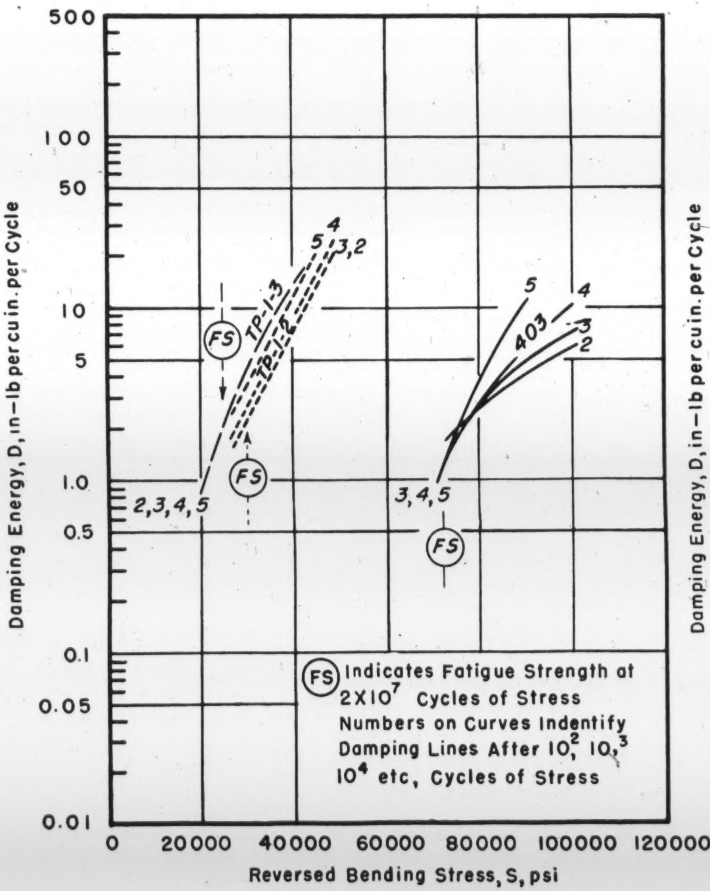

FIG. 26.—*D-S-N* Diagrams for 403, TP-1-2, and TP-1-3 Comparing Damping Energy at 500 F as a Function of Stress History and Magnitude.

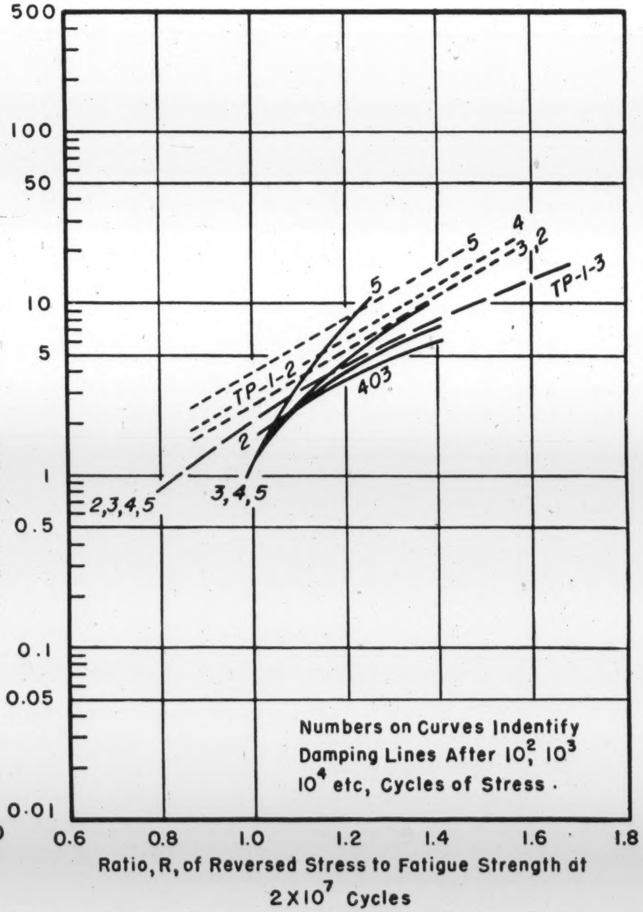

FIG. 27.—*D-R-N* Diagrams for 403, TP-1-2, and TP-1-3, Comparing Damping Energy at 500 F as a Function of Stress History and Stress Ratio.

ratios of 1.0 than do the dynamically weaker materials.

A third factor not mentioned in this discussion of the comparison of damping properties is the effect of stress history. The hypothetical curves drawn in Fig. 23 apply to a fixed stress history. However, one material might have decreasing damping values with increasing number of cycles at stress while another might have the opposite trend so that a given material could be inferior to another after 100 cycles of stress, yet be considerably superior after exposure to, say, 100,000 cycles.

A comparison of materials tested in this program on the basis of the same stress magnitude is given in Figs. 24, 26, and 28. These graphs are similar to the upper diagram in Fig. 23. They show damping energy, *D*, plotted to a log scale as a function of reversed bending stress, *S*, with iso-history contour lines connecting the damping values after a given number *N* of reversed constant stress cycles. They are termed *D-S-N* diagrams. Similar graphs comparing the damping of materials on the basis of stress ratio rather than stress magnitude are termed *D-R-N* diagrams. The *R* indicates the ratio of the reversed stress to the fatigue strength of the material. Diagrams of this type for the materials tested in this program are shown in Figs. 25, 27, and 29.

A comparison of the room temperature damping properties on the basis of the same stress magnitude is presented in Fig. 24 for all of the materials tested at room temperature. The fatigue strength at 20 million cycles of stress is indicated for each material by a flag marked F.S. Iso-history contour lines are marked by 2, 3, 4, etc., to indicate the damping after 10^2, 10^3, 10^4, etc., stress cycles. The data for all materials except Inconel X were obtained using type E specimens. For that material a larger specimen was employed as described in the discussion of the *S-N* curves. It is observed in Fig. 24 that the relative damping energy values among a group of materials at a given stress vary in a general way inversely as their fatigue strengths. For example, the TP-1 materials have the lowest fatigue strengths; yet at a given stress value, they display greater damping than the dynamically stronger materials. Inconel X is one major exception to this behavior for, although possessing an intermediate value of fatigue strength, its damping at a given stress is about the lowest of all the materials. It is improbable that the larger size specimens used for Inconel X can explain this nonconformity.

The effects of stress history on the damping properties of the various materials are also apparent from this diagram. Thus, materials TP-1-2, TP-2-R, TP-2-B, and type 403 are seen to have generally increasing damping with an increase in number of cycles at constant stress; S-816 displays the reverse tendency; the damping of TP-1-3 as noted previously is unaffected by stress history; and Inconel X displays a combination of the above trends. The room temperature damping properties of TP-2-R and Inconel X are more affected by stress history than those of the other materials.

The damping properties of the same seven materials at room temperature are compared on the basis of stress ratio by means of the *D-R-N* diagrams of Fig. 25. Many of the features discussed in connection with the lower diagram of Fig. 23 are displayed by these groups of curves. The damping of type 403 which is inferior to that of most of the other materials on the basis of the same stress (Fig. 24) is seen to be many times greater than that of any of the other materials except S-816 when compared at a stress ratio of one. Alloy S-816 displays unusual properties in that its damping capacity com-

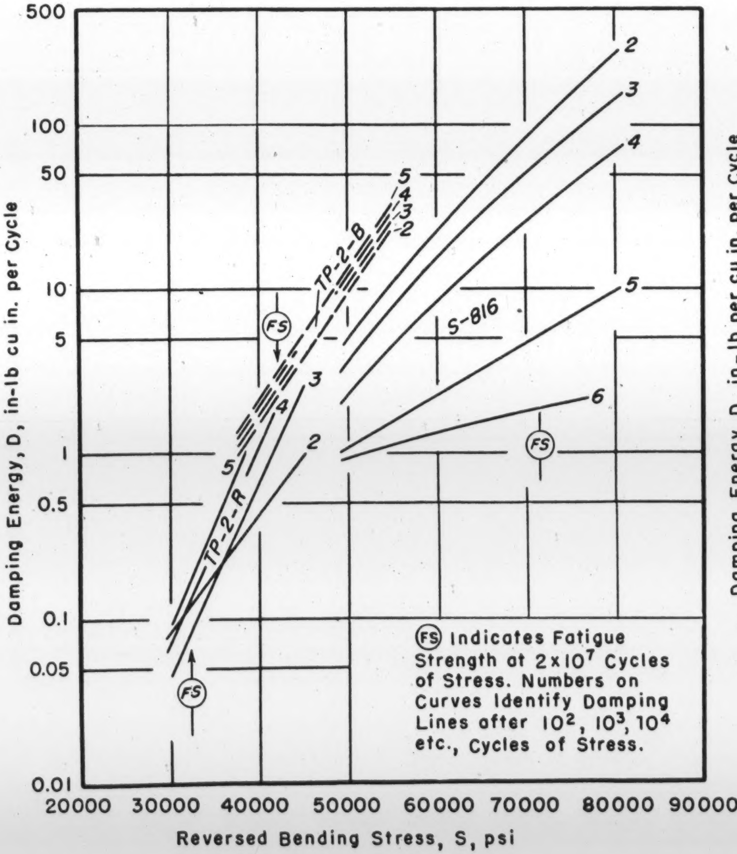

FIG. 28.—*D-S-N* Diagrams for S-816, TP-2-R, and TP-2-B, Comparing Damping Energy at 900 F as a Function of Stress History and Stress Magnitude.

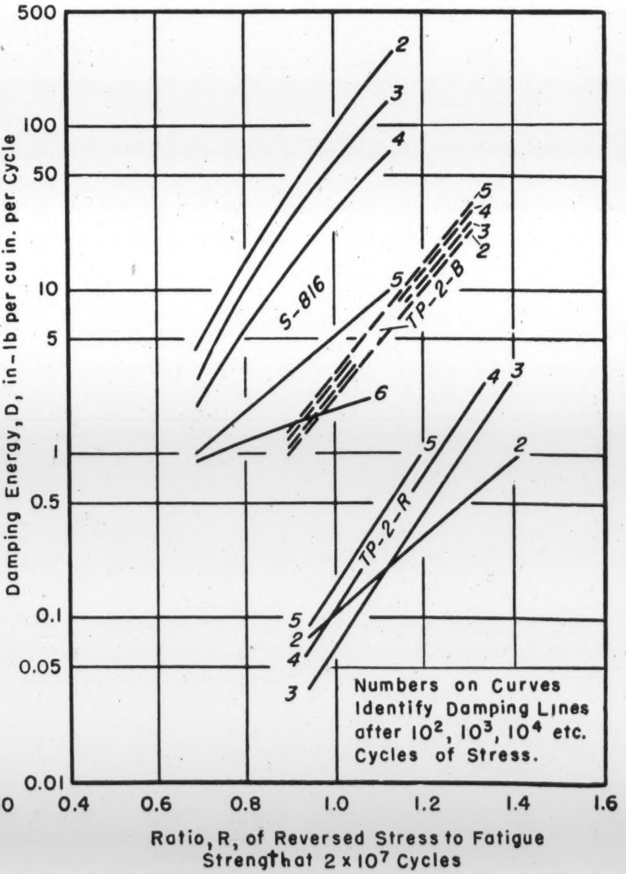

FIG. 29.—*D-R-N* Diagrams for S-816, TP-2-R, and TP-2-B, Comparing Damping Energy at 900 F as a Function of Stress History and Stress Ratio.

pares favorably with the other materials on the basis of equal stress and is also superior to all of those tested (except type 403 after a large number of cycles) when compared on the basis of the same stress ratio. The TP-1 materials have a tendency to display damping values superior to all of the other materials tested for low stress ratios. The effect of different heat treatments on the same powder metal is shown by comparing the curves for TP-1-2 with those for TP-1-3. It is also interesting to compare the curves for TP-2-B, the arc-cast molybdenum plus 2 per cent tungsten material, with those for TP-1-2, sintered iron powder. They have similar damping values for a stress ratio of one, yet the curves have sharply different slopes. The combined effects of a different method of manufacture and a difference in composition are seen in comparing the curves for TP-2-B and TP-2-R.

The relative order of merit for the damping properties of a group of materials may be the same at elevated temperature as at room temperature or it may be considerably different. Obviously, then, the damping of materials should be determined at the temperature of service. In comparing the room temperature damping properties of the three materials, type 403 stainless (extrapolated to low stress values), TP-1-2, and TP-1-3 on the basis of magnitude of reversed stress (see Fig. 24), the two powder metals display considerably higher damping at a given stress. As seen in Fig. 26, the same general relationships hold true for the properties at 500 F with the damping values for the powder metals being considerably higher than those for the wrought type 403 for stresses in the range of 20,000 to 50,000 psi. After 100,000 cycles of stress, the curves for the two TP-1 materials are very close to each other.

When the same three materials are compared on the basis of stress ratio, however, as in Fig. 27, it is seen that the values for the wrought and powder metals are much closer together. For a large number of stress cycles, the damping values for TP-1-2 are greater than those of TP-1-3 at all stress ratios. Both of these materials exhibit somewhat higher damping values than type 403 at a stress ratio of one, but after a large number of stress cycles at the higher stress ratios, the damping of type 403 becomes the greatest.

Figures 28 and 29 compare the 900 F damping properties of materials S-816, TP-2-B, and TP-2-R. On the basis of the same magnitude of applied stress, Fig. 28 indicates that TP-2-B absorbs greater energy than either of the other two materials at stress values below 40,000 psi. Due to the greater rate of change of damping with stress, TP-2-R displays higher damping values after a large number of stress cycles at stresses greater than about 40,000 psi than either S-816 or TP-2-B. The relative effects of stress history on the damping properties of the three materials are apparent from this plot. S-816 is affected far greater by stress history than either of the other two materials.

Since the fatigue strengths of the three materials tested at 900 F vary widely, the plot of damping as a function of stress ratio shown in Fig. 29 is an interesting one. If a comparison is made at a stress ratio of one, S-816 after 100 cycles of stress displays a damping energy about 40 times that of TP-2-B and about 1000 times that of TP-2-R. After 100,000 cycles of stress, however, the damping of S-816 is less than twice that of TP-2-B and only about 25 times that of TP-2-R. After a greater number of cycles of stress, the damping of S-816 falls below that of TP-2-B, but is still greater than that of TP-2-R. At values of stress ratio less than about 0.8, S-816 displays higher damping than either of the other two materials.

No attempt is made in this paper to express mathematically the relationships among damping energy, stress, and stress history. It should be mentioned however, that logarithmic plating of damping *versus* stress result reasonably straight lines in many cases and damping may be related to stress by the expression:

$$D = QS^n$$

where Q and n are constants. The range of exponent n for the materials tested is from 2 to 30.

Comparison of the Elasticity Properties:

In Fig. 30 are presented E_d - S-N diagrams for six of the materials tested at room temperature. This graph shows dynamic modulus of elasticity E_d as a function of reversed bending stress, S, with lines connecting the dynamic modulus of elasticity values after a given number N of reversed constant stress cycles. The horizontal dashed lines indicate the static modulus values for the virgin specimens. The F.S. flags have their usual meaning. It should be noted that for room temperature the differences between the dynamic moduli and the static moduli are small for stresses up to the fatigue strength of a material. S-816 displays the greatest decrease in modulus value at the fatigue strength in which case after 100 cycles of stress there is a decrease of somewhat less than 3 per cent from the virgin static value. At stresses in excess of the fatigue strength of a given material, the decrease in stiffness is in some cases appreciable. For example, for TP-2-R after 100,000 cycles at a stress approximately 1.5 times its fatigue strength, there is a decrease to about 90 per cent of the initial static modulus. Even this 10 per cent decrease is considerably smaller than that observed for mild steel (1), which is 35 per cent at a stress ratio of 1.16. Stress history seems to have small effect on the dynamic modulus of TP-1-3 and perhaps the greatest effect on that of TP-2-R.

Figure 31 shows the E_d-S-N diagrams for the same six materials at elevated temperatures: 500 F for the TP-1 materials and type 403; 900 F for the TP-2 materials and S-816; and 1600 F for S-816. The method of plotting is identical with that of Fig. 30. The dynamic moduli values at stresses corresponding to the elevated temperature fatigue strengths are only a few per cent different (usually lower) than the virgin static moduli at the same temperature for all materials and all temperatures investigated except in the case of S-816 at 900 F. As pointed out previously in connection with Fig. 9, both stress magnitude and stress history exert a large effect on the dynamic modulus of S-816 at this temperature. For example, to show the effect of stress history, at a stress corresponding to the 900 F fatigue strength of S-816, the dynamic modulus after 100 cycles of stress is about 80 per cent of the initial static modulus. After 1,000,000 cycles of stress, the dynamic modulus value is about 102 per cent of the initial static value. Observing the effect of stress magnitude, it is seen that at a stress of 50,000 psi after 100 cycles the dynamic modulus has a value about 98.5 per cent of the initial static value while at 80,000 psi after the same number of cycles its value is only 71 per cent of the initial static value. Both stress magnitude and stress history exert an appreciable effect on the dynamic modulus of the TP-2 materials at 900 F for stresses considerably in excess of the fatigue strengths of the materials. The effects of these two variables are less pronounced in the case of the materials tested at 500 F. Material TP-1-3 remained unaffected by stress history at 500 F, a behavior similar to that displayed at room temperature at low stress.

The absolute value of the modulus of

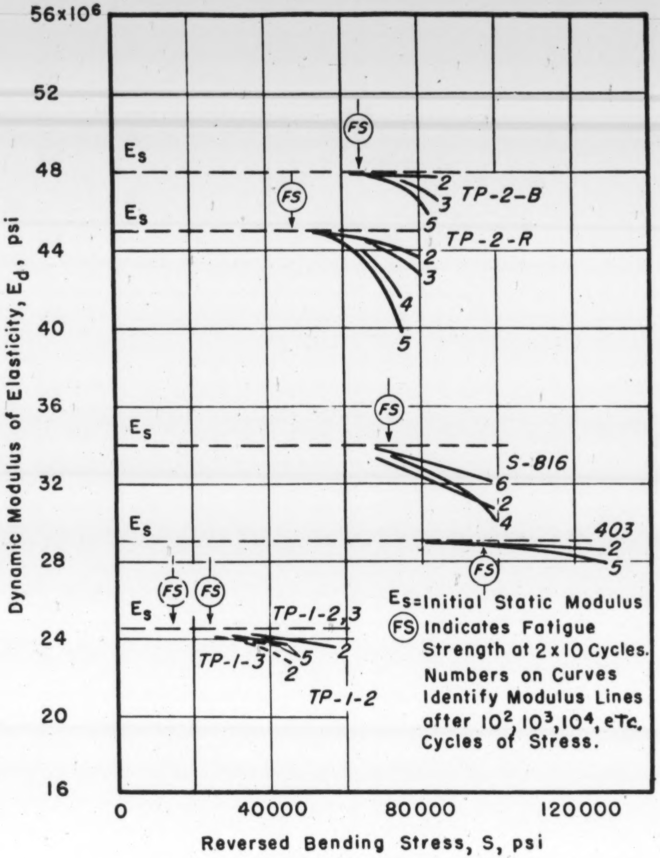

FIG. 30.—E_d-S-N Diagrams for Several Temperature-Resistant Materials Comparing Dynamic Modulus of Elasticity at Room Temperature as a Function of Stress History and Stress Magnitude.

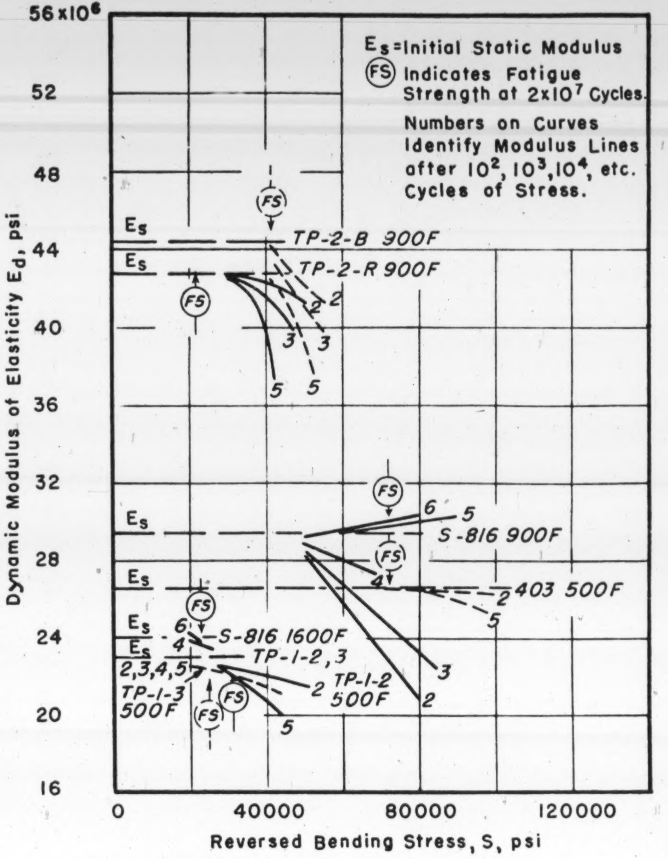

FIG. 31.—E_d-S-N Diagrams for Several Temperature-Resistant Materials Comparing Dynamic Modulus of Elasticity at Elevated Temperatures as a Function of Stress History and Stress Magnitude.

elasticity of a material at operating temperature is a major factor in determining the natural frequency of a blade made of that material. The use of the natural frequencies in machine design was discussed previously in this paper. For a very rough comparison of materials, it may be assumed that as a first approximation the natural frequency is proportional to the square root of the modulus of elasticity of the material. The relative frequencies of a given blade made of different materials may then be compared by using the second roots of the moduli of elasticity of the materials assuming the masses of the different blades to be the same. Such a comparison may be made among the materials tested at the same temperature by using the square roots of the static values of elastic modulus given in Table II. This table gives the static moduli of elasticity for the materials tested at the various temperatures. The static values may be used since relatively little change from these values was observed with cyclic stress history for stress values near the fatigue strengths of the materials. The one major exception to this is, of course, S-816 at 900 F which even at a stress equal to the fatigue strength has dynamic modulus values displaying major deviations from the initial static modulus. The natural frequency of a blade made from this material and subjected to a cyclic stress equal to its fatigue strength would, at this temperature, be affected greatly by the stress history of the blade.

TABLE IV.—COMPARISON OF APPROXIMATE RELATIVE AMPLITUDES AT RESONANCE.

Material	Cyclic Stress to Produce Damping of 5 in-lb per cu in. per cycle After 10^5 Cycle of Stress, psi	E_d at 10^5 Cycles, psi	Unit Strain, S/E_d, in. per in.
Room Temperature			
S-816	70 000	33 700 000	0.00208
TP-2-R	67 500	43 100 000	0.00156
TP-2-B	78 000	46 900 000	0.00166
Type 403	80 500	28 800 000	0.00279
TP-1-2	43 000	23 700 000	0.00181
TP-1-3	36 500	23 800 000	0.00153
500 F			
Type 403	82 000	26 300 000	0.00311
TP-1-2	33 000	21 900 000	0.00151
TP-1-3	31 000	22 100 000	0.00140
900 F			
S-816	71 000	29 800 000	0.00238
TP-2-R	44 500	34 000 000	0.00131
TP-2-B	44 000	41 900 000	0.00105

It would be impossible to determine the desirability of one material with respect to another as far as the relative blade stiffness properties are concerned unless machine design data were available and natural frequency calculations (9) made for blades of each material. With this information the location of the critical frequency spectrum, with respect to the operating speed range of the machine, would be known for blades of each material. For the frequency calculations, elevated temperature moduli should be used. Since the temperatures vary at different stations along the length of the blade, the modulus values should be known for a range of elevated temperatures. The variation of the dynamic moduli with stress and with stress history for the different materials as given in Figs. 30 and 31 will afford an indication of the amount of change that can be expected in the natural frequency of a given blade made of various materials when operating under various conditions at the temperatures indicated.

The last consideration to be discussed relative to stiffness properties is that of a comparison among a group of materials for the amplitude of vibration when passing through or operating at a resonant frequency. This comparison should be made on the basis of equal energy dissipation since materials of differing damping properties must be subjected to cyclic stress of different magnitudes to produce the same energy dissipation. These stresses produce different values of deflection in blades made of various materials depending on the elasticity properties of those materials. Such a comparison is presented in Table IV. The data for column 2 of this table were obtained from Fig. 5 for S-816 and similar *D-S-N* plots for the other materials. The value of 5 in-lb per cu in. per cycle and the number of stress cycles (10^5) were arbitrarily chosen to obtain values for comparison. The E_d values of column 3 were read from large scale E_d-*S-N* diagrams similar to Figs. 30 and 31. The values in column 4 may be used to determine the relative amplitudes of deflection for similar blades made from different materials. Thus, at a resonance for which the vibrational energy input to the material itself is 5 in-lb per cu in per cycle, the deflection at 500 F of a blade made of type 403 would be more than twice that of a blade made of TP-1-2. Similarly, a blade made of S-816 would have a deflection at resonance greater than twice that of one made of TP-2-B when dissipating the same amount of energy at 900 F. It is also interesting to note that the unit strains (or relative amplitudes) of S-816 and type 403 increase upon going from room temperature to an elevated temperature whereas the values for the other four materials have the opposite trend. In applications where deflections are limited by the design of the machine, then such factors as discussed above are important in materials selection.

TABLE V.—QUALITATIVE RATINGS[a] OF MATERIALS AT ROOM AND ELEVATED TEMPERATURES.

Material	Fatigue Strength		Damping Capacity for Stress Ratio of 1.0		Least Deflection at Resonance for Blade of a Given Material	
	RT	500 F	RT	500 F	RT	500 F
Type 403	1	1	1	3	3	3
TP-1-2	2	2	2	1	2	2
TP-1-3	3	3	3	2	1	1
	RT	900 F	RT	900 F	RT	900 F
S-816	1	1	1	1 or 2[b]	3	3
TP-2-R	3	3	3	3	1	2
TP-2-B	2	2	2	1 or 2[b]	2	1

[a] 1, 2, and 3 indicate decreasing order of merit with respect to a given property.

[b] Rating is dependent upon stress history.

The comparison of resonant vibration amplitude discussed above is important not only in considering clearance and other design factors, but it may also indicate the volume of noise produced during resonant conditions. Thus, column 4 of Table IV may be significant in selecting materials for applications where noise must be kept at a low level.

Materials Selection:

In previous sections, the fatigue, damping capacity, and modulus of elasticity properties for groups of materials have been compared and evaluated separately in a quantitative fashion. A qualitative rating of the materials in a group according to the three properties for the two test temperatures is shown in Table V to facilitate materials selection. Table V was prepared from Figs. 22 through 31. It is obvious that while room temperature tests may indicate the relative rating at elevated temperatures with respect to one property, they may be misleading with respect to other properties.

For the final evaluation of materials for a given application, a table such as Table V can be set up with other columns for properties which also affect service behavior such as creep, resistance to oxidation, ductility, etc. Each material could be rated separately according to each property and then, if the relative importance of these properties is known for a given application, a final over-all figure of merit for each material could be determined. This index of serviceability, considered along with availability, cost, workability, etc., would govern materials selection.

SUMMARY AND CONCLUSIONS

Damping capacity, dynamic modulus of elasticity, and fatigue strength properties are of great importance in the design of parts used in applications involving dynamic loads. Data are reported in this paper on the results of room and elevated temperature tests designed to reveal changes in the damping and elasticity properties of various materials during constant reversed cyclic stress tests at engineering stress levels. Newly developed rotating-cantilever beam fatigue-testing equipment was used in this work.

Data for S-816 at room temperature 900 F, and 1600 F; for TP-2-R and TP-2-B at room temperature and 900 F; for type 403 TP-1-2, and TP-1-3 at room temperature and 500 F; for Inconel X at room temperature; and for low carbon N-155 at 1500 F, presented in a series of new diagrams, show the dependence of the damping and elasticity properties upon both stress magnitude and stress history and facilitate comparison and evaluation of these properties. Various patterns have been observed in the behavior of these properties during constant stress tests carried out at various temperatures. For all materials and at all temperatures investigated, the energy dissipated by damping increases rapidly with stress at values close to the fatigue strength of a material. During a constant reversed cyclic stress test, the damping energy may decrease, remain the same, increase, or have a varying pattern as the number of stress cycles is increased. In general, the changes in dynamic modulus of elasticity are reciprocal to the changes in damping energy.

Room and elevated temperature *S-N* data presented for the various materials indicate that, in general, the curves tend to become flatter as the temperature level is raised. The elevated temperature fatigue strengths for temperature resistant materials may be lower, equal to, or greater then the room temperature values.

Two methods for comparing the damping and elasticity properties of materials are presented and the merits of each discussed. The first is on the basis of equal stress magnitude and the second is on the basis of the same ratio of applied stress to fatigue strength. The latter method should be employed in applications where resonant vibrations are encountered since the service requirement is a given amount of energy absorbed rather than a definite stress applied. Special diagrams presented facilitate a comparison of the damping properties for each of the methods outlined above. Evaluations of the damping properties are made for the groups of materials tested at the same elevated temperatures.

Figures 30 and 31 for room temperature and elevated temperature tests facilitate comparison of the elasticity properties. They show the dependence of the dynamic modulus of each material upon stress magnitude and stress history. The square roots of the moduli of elasticity reported in Table II provide an indication of the approximate relative natural frequencies of vibration of turbine or compressor blades made of the various materials. Table IV shows the approximate relative deflections obtained at resonance for blades made of various materials.

Table V outlines a method for comparing different materials on the basis of their dynamic properties: damping, elasticity, and fatigue strength.

In conclusion, it is felt that the new diagrams and tables for explanation and evaluation of the behavior of the dynamic properties of materials and the new and revealing data presented may aid in the comprehension of one of the least understood of the dynamic properties, damping, and emphasize the large changes which may take place in another, dynamic modulus of elasticity. It is hoped that the methods for the comparison and evaluation of materials will be of value either in selecting a material for a new application, or for rating replacement materials by comparing their properties with those of materials which have already proved their worth in a given application.

Acknowledgment:

The fine cooperation and encouragement of the Office of Naval Research, Thompson Products Corp. and the U. S. Air Force, sponsors of this work, are greatly appreciated.

All of the test specimens except N-155 were furnished in the form for final polishing by Thompson Products Corp. Specimens of N-155 were furnished in final form by the National Advisory Committee for Aeronautics.

S. Tomkinson, G. Gronau, M. Linza, and P. Madonna assisted in the development of the machines and in the preparation and testing of the specimens.

REFERENCES

(1) B. J. Lazan, "A Study With New Equipment of the Effects of Fatigue Stress on Damping Capacity and Elasticity of Mild Steel," *Transactions*, Am. Soc. Metals, Vol. 4, pp. 499–558 (1950).

(2) J. M. Robertson and A. J. Yorgiadis, "Internal Friction in Engineering Materials," *Journal of Applied Mechanics*, Vol. 13, pp. A173–A181.

(3) B. J. Lazan and A. J. Yorgiadis, "The Behavior of Plastics Under Repeated Stress," Symposium on Plastics, Am. Soc. Testing Mat., pp. 66–94 (1944).

(4) B. J. Lazan and T. Wu, "Damping, Fatigue, and Dynamic Stress-Strain Properties of Mild Steel," p. 649, this publication.

(5) I. Perlmutter, "Service Failures of Turbine Buckets," Air Force Technical Report No. 5716, July, 1948.

(6) B. J. Lazan, "Some Mechanical Properties of Plastics and Metals Under Sustained Vibrations," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 65, pp. 87–104 (1943).

(7) O. Föppl, "The Practical Importance of Damping Capacity of Metals, Especially Steels," *Journal*, Iron and Steel Inst., Vol. 134, pp. 393–455 (1936).

(8) B. J. Lazan, "Dynamic Creep and Rupture Properties of Temperature-Resistant Materials Under Tensile Fatigue Stress," *Proceedings*, Am. Soc. Testing Mats., Vol. 49, pp. 757–798 (1949).

(9) A. Herzog, "Calculations of Natural Frequencies and Stresses, and Proposed Testing Methods," Air Force Technical Report No. 5936, Part I, May, 1950.

DISCUSSION

Chairman T. J. Dolan.[1]—I should like to ask a question, with respect to damping. I noted in some of the later figures that the values became considerably larger. If we select a rough value from the curves of about 12-in-lb per cu in. per cycle and assume that we have, let us say, a turbine blade of 1 cu in. of material vibrating at 1000 cps, a rough computation indicates that this is about equivalent to dissipation of 2 hp. On a 1-cu in. specimen this will cause considerable heating. I wonder whether this of itself might not throw the data from, let us say, the 900 F curve to the 1600 F curve and thus cause difficulty in predicting what the operating condition in the blade might actually be under this type of severe testing.

Mr. E. H. Schuette.[2]—The curves show what is, to me, an astonishing variation in dynamic modulus with cycles. Is it the elastic modulus that was recorded, or perhaps simply the secant modulus between the end points of the stress-strain loop?

Chairman Dolan.—I assume, that in these measurements of damping the authors have utilized the lateral deflection of the rotating beam, and that the measurements of the dynamic modulus are measurements of the relative vertical deflection of the specimen under load during the test?

Mr. B. J. Lazan (*author's closure*).—Mr. Dolan has inquired about the magnitude and implications of the temperature increases during a cyclic stress test as a result of internal damping.

As many in the fatigue testing field have observed, the temperature increase caused by damping may be very large. For example, in a recent room-temperature fatigue test on N-155 alloy under direct stress near the fatigue limit at 3600 cycles per min the test specimen attained a temperature of approximately 1500 F within three minutes after the start of the test. In most cases, however, the temperature increase in much smaller than cited above.

In calculating temperature increases from damping data, one must consider the frequency sensitivity of damping capacity. A companion paper (**4**)[3] indicates that for mild steel at least, the damping in the high-stress region decreases with frequency beyond approximately 1 cps. Other materials now under study at the University of Minnesota display a similar trend. One should therefore be cautious in using low-frequency damping data to determine energy dissipation and consequential temperature increases at high frequency. The 2 hp dissipation per cu in. at 1000 cps computed by Mr. Dolan may therefore be much too large.

A second factor which would tend to keep the temperature lower than indicated by the damping data presented is that actual parts rarely receive uniform stress in service: generally speaking there are a few regions of high stress at discontinuities, etc., and most of the part is at relatively low stress. Thus, the volume of metal at peak stress is generally quite small in an actual part, and furthermore the low-stress regions surrounding the high-stress locations effectively transmit and dissipate the heat. Both of these factors tend to keep the operating temperature of a part lower than one might imply from damping data.

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[2] Division of Tests, Metallurgical Labs., The Dow Chemical Co., Midland, Mich.

[3] The boldface numbers in parentheses refer to the list of references appended to the paper, see p. 646.

Now what are the implications of the temperature increases resulting from damping. As Mr. Dolan points out, the operating temperature of a part may be significantly higher than the environmental temperature, with a resulting weakening of the part. This temperature difference must of course be considered in strength and life predictions. This difference must also be considered in laboratory fatigue tests. In the laboratory test it is sometimes necessary to adjust the environmental temperature so that considering the temperature increase caused by damping the specimen will be at the desired testing temperature. It is also necessary to consider temperature distribution within the specimen in the design of such an experiment.

In answer to Mr. Dolan's questions concerning testing procedures, the method used for measuring damping involved the lateral deflection of a rotating beam. This method is described in earlier papers (1) and (4).

Messrs. Dolan and Schuette inquire about the definition of the dynamic modulus of elasticity as used in this paper. As explained in the early papers (1) and (4), the equipment enables measurement of the ratio of the maximum moment on the specimen to its maximum deflection. From this may be calculated the ratio of maximum stress to strain. Hence, the dynamic modulus given is a secant value between zero and maximum stress. An analysis of the factors affecting this change in dynamic modulus is discussed in the companion paper (4) and its implications are discussed in an earlier paper (6).

DAMPING, FATIGUE, AND DYNAMIC STRESS-STRAIN PROPERTIES OF MILD STEEL*

BY B. J. LAZAN[1] AND T. WU[2]

SYNOPSIS

Damping energy, dynamic modulus of elasticity, and fatigue properties of mild steel are investigated in recently developed rotating-beam testing equipment. Data on the effect of several important test variables, such as stress magnitude, history, frequency, and rest are presented. Stress magnitude and stress history data are analyzed in terms of cyclic stress sensitivity limit and stabilized damping points. These data are presented in *S-N-N*, *S-N-D*, and other new types of diagrams to indicate not only the fatigue behavior but also the damping and dynamic modulus properties. The effect of frequency of cyclic stress on damping and dynamic modulus is found to be considerable at stresses above the dynamic proportional limit. Dynamic stress-strain data are presented and analyzed to indicate the effect of stress magnitude and stress history on the dynamic proportional limit. Frequency sensitivity data are also presented in terms of strain rate and flow stress.

Initial exploratory work on a study and correlation of the damping, fatigue, and elastic modulus properties of mild steel was discussed in a recent paper (**1**)[3] by the senior author. In this work the damping energy and dynamic modulus of elasticity of a specimen were continuously measured with newly developed rotating beam equipment during a fatigue test. Thus damping and elasticity properties were procured as a function of not only the amplitude of reversed stress but also another equally important variable, stress history.

The research reported in this paper not only extends the earlier work on mild steel but also analyzes the damping and modulus properties in terms of dynamic stress-strain behavior. Also presented are data on the effects of frequency of cyclic stress, its analysis in terms of strain rate, and its relationship to the cyclic plastic strain.

This work is part of a broad exploratory program on the inelastic behavior of materials at dynamic stress levels of engineering significance, that is, stresses up to approximately 150 per cent of the fatigue limit of materials. In its broader aspects, this inelastic behavior study includes not only the correlation among damping capacity, dynamic modulus of elasticity, fatigue, and dynamic stress-strain characteristics, but also the relationship of these properties to creep, stress relaxation, and other properties associated with the inelasticity of materials.

This paper presents experimental data on the inelastic behavior of mild steel under cyclic stress and undertakes some applied mechanics interpretation of these

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* Presented at the Fifty-fourth Annual Meeting of the Society, June 18–22, 1951.

[1] Professor of Materials Engineering, University of Minnesota, Minneapolis, Minn.; Formerly at Syracuse University, Syracuse, N. Y.

[2] Hong Kong, China; Formerly at Syracuse University, Syracuse, N. Y.

[3] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 678.

data. Discussion and analysis of these inelastic properties in terms of physics of metals theory are beyond the scope of this paper.

TEST EQUIPMENT

The rotating-beam dynamic testing machine developed and used in this work is described in a previous paper (1). However, since several changes have recently been made in this machine and in the interest of completeness the latest model of the machine is briefly described below.

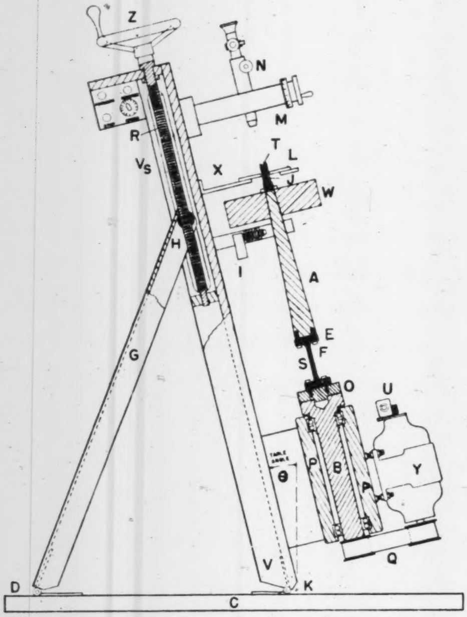

FIG. 1.—Schematic Diagram of Rotating Cantilever Beam Dynamic Testing Machine.

The principle of operation of the rotating cantilever-beam dynamic testing machine may be understood from the schematic diagram and photograph shown in Figs. 1 and 2. Test specimen S is clamped at its bottom end to cap O and loaded through its top end by extension arm A and loading weight W. The arm-specimen-cap assembly is supported by an extremely accurate rotatable spindle B. This spindle is supported by table V, rotatable on hinge K to any desired angle, θ, which is steplessly adjustable by screw R. If table V is verticle (angle $\theta = 0$ deg) the combined weight of arm A and weight W produces no bending moment in the test specimen S. Under this condition, the alternating stress produced in the specimen during rotation of the spindle and attached parts is zero. Since the static compressive stress due to weight of parts is insignificant, the speci-

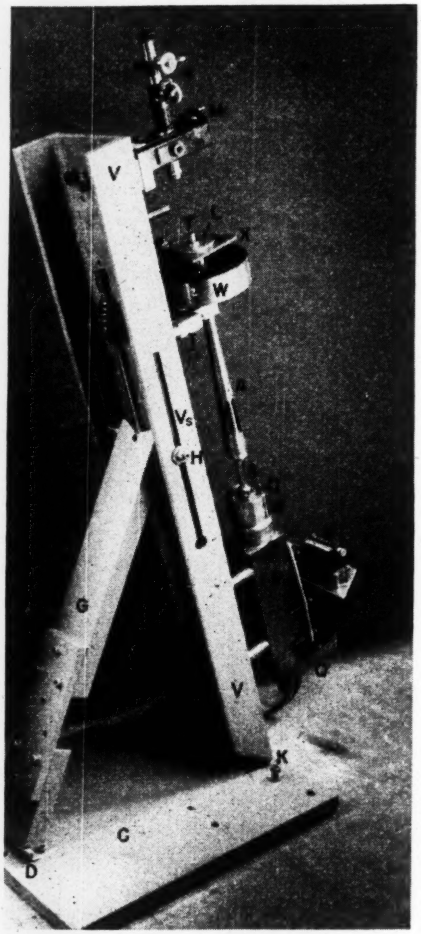

FIG. 2.—Photograph of Rotating Cantilever Beam Dynamic Testing Machine.

men may be considered to be at zero stress. Since the bending moment is proportional to the sine of table angle, θ, the stress in the specimen may be steplessly adjusted to the desired value by handwheel *Z*.

A damper system *L*-*X* is incorporated to minimize the vibration of arm *A* and to facilitate accurate measurement of the position of target *T* at the free end of the extension arm. The micrometer slide *M* and measuring microscope *N* assembly are used to measure both coordinates of the position of the target. As discussed in the previous paper (1) on this work the dynamic modulus of elasticity and the damping energy may be calculated in accordance with the following equations:

$$D = K\,(SH) \quad \text{(1)}$$

and

$$E_d = C\,(S/V) \quad \text{(2)}$$

where:

D = damping energy absorbed by the specimen in in-lb per cu in. per cycle of stress,

E_d = dynamic modulus of elasticity (secant value) during reversed bending stress, psi,

S = amplitude of reversed stress in specimen, psi,

V = vertical (gravitational) deflection of target *T* (toward *X* in plane of Fig. 1) due to bending stress in specimen, in.,

H = horizontal traversal of the target *T* or its total lateral displacement (perpendicular to direction of its gravitational deflection) caused by reversing the direction of rotation of the spindle, in., and

K and C = constants dependent only on specimen shape, weight and center of gravity of loading arm assembly, and the location of the target along the axis of the arm.

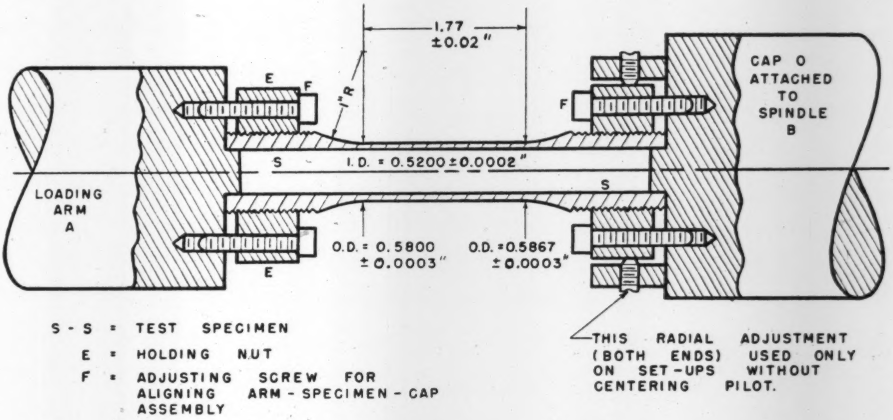
1.77 ±0.02"
1"R
E
F
CAP O ATTACHED TO SPINDLE B
F
S
I.D. = 0.5200 ± 0.0002"
S
LOADING ARM A
E
O.D. = 0.5800 ± 0.0003"
O.D. = 0.5867 ± 0.0003"
S - S = TEST SPECIMEN
E = HOLDING NUT
F = ADJUSTING SCREW FOR ALIGNING ARM - SPECIMEN - CAP ASSEMBLY
THIS RADIAL ADJUSTMENT (BOTH ENDS) USED ONLY ON SET-UPS WITHOUT CENTERING PILOT.

Fig. 3.—Shape of Test Section for Type P Specimen and Method of Gripping.

The method of gripping the test specimen is shown in Fig. 3. Nuts *E*, screwed on the ends of the threaded test specimen and clamped in place by screws *F*, rigidly fix the specimen to cap *O* and extension arm *A*. The entire arm-specimen-cap assembly may be very accurately aligned on dead centers before being placed in the machine by preferentially tightening adjusting screws *F*. Furthermore, screws *F* preload the entire joint so as to minimize backlash and extraneous energy losses, a very important consideration in damping work. Radial screws *R* are used only for setups in which a centering pilot

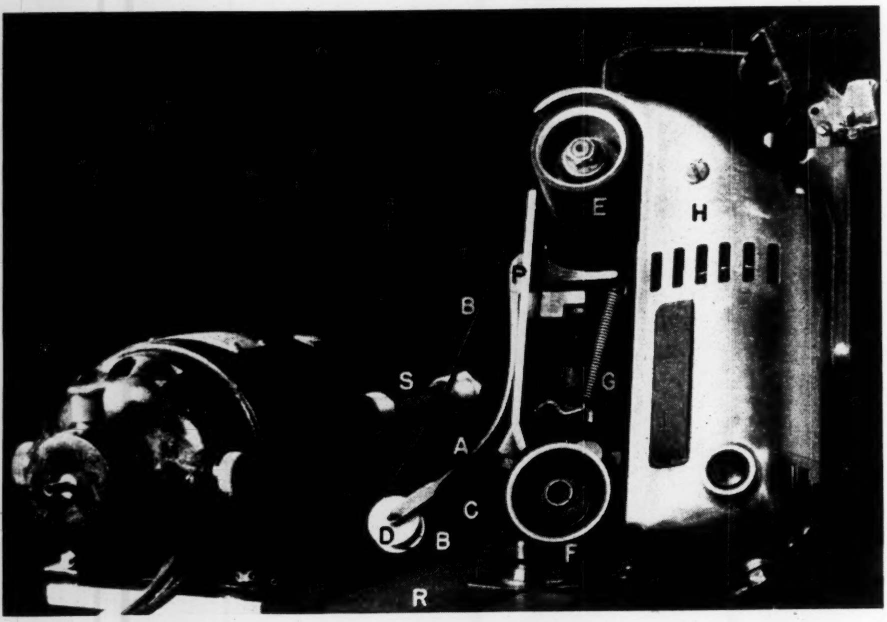
E
H
P
B
S
G
A
C
D
B
F
R

FIG. 4.—Photograph of Controlled-Pressure Belt Polisher.

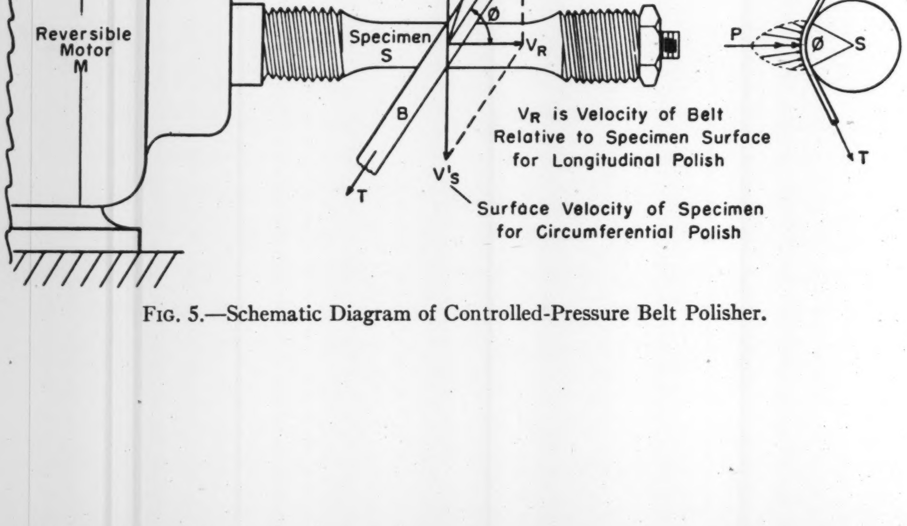
V'R is Velocity of Belt Relative to Specimen Surface for Circumferential Polish
T–Tension in Belt
V'R
B–Polishing Belt
VS is surface Velocity of Specimen for Longitudinal Polish
VS
VB–Linear Velocity of Belt
Reversible Motor M
Specimen S
Ø
VR
B
T
V'S
VR is Velocity of Belt Relative to Specimen Surface for Longitudinal Polish
Surface Velocity of Specimen for Circumferential Polish
P
Ø
S
T
T

FIG. 5.—Schematic Diagram of Controlled-Pressure Belt Polisher.

is not used. This holding fixture is not only simple and effective but also permits gripping the specimen through simple threaded ends requiring only standard manufacturing tolerances.

Further details on machine design, testing techniques, and method of reducing the experimental data are discussed elsewhere (1).

TEST SPECIMEN AND ITS PREPARATION

The test section of all specimens used in this work is shown in Fig. 3. The justification for using this hollow tapered specimen, in preference to the more easily made solid specimen, is discussed in the earlier paper (1).

Most of the test specimens were finished by the contour block-belt sanding method used in previous work (2). Although this method of specimen finishing produced a good surface finish with a longitudinal polish (existing polishing scratches parallel to direction of normal stress), there was some evidence that, in spite of all precautions, limited surface buffing, "cold-working" or "smear," could not be avoided. Such buffing not only changes the properties of the all-important metal at the surface but also makes it difficult to produce specimens of the desired uniformity. Damping properties are particularly sensitive to this buffing action. It was concluded that in order to minimize surface buffing not only sharp sanding belts were required to assure cutting rather than smearing action but also the pressure between the belt and the specimen must be controlled. Attempts to control the belt pressure through use of resilient layers between the contour block and the polishing belts were not wholly successful. Therefore, a new controlled-pressure, belt polishing method was developed.

The principle of operation of the new controlled-pressure belt polisher may be understood from the photograph and schematic diagram shown in Figs. 4 and 5. Referring to Fig. 4, test specimen *S* is held by an arbor which is rotated by reversible motor *M*. Polishing belt *B* is driven and guided by pulleys *D*, *E*, and *F*, which are part of sander assembly *H*, and contacts specimen *S* over a small arc as shown. Pulley *D* is held by arm *A* which is pivoted at *P* and loaded by spring *G*, the force in which is adjustable by means of screw *C*. Thus, the tension in the belt is adjustable by screw *C* and maintained by flexible spring *G*. Since table *R* has a reciprocating motion between two adjustable stops, the entire length of the test section, including fillets, may be polished.

The relationship of the sanding belt *B* to specimen *S* is shown in Fig. 5. The belt wraps around the specimen through the angle ϕ and produces a pressure distribution approximately as shown, which has a resultant *P*. This pressure distribution is a function of (*a*) the belt tension *T*, (*b*) the contact angle ϕ, and (*c*) the angle θ between the belt and the axis of the specimen, all of which are readily adjustable.

Angle θ between the specimen and the belt is less than 90 deg in order to control the direction of polishing as explained below. Assume that the front of the specimen, as viewed in Fig. 5, has a surface velocity, V_S, upward as shown. Assume, also, that the belt has a velocity V_B in the direction shown. Under these conditions the velocity of the belt *relative* to the specimen is the vector difference between V_S and V_B, which is V_R. This relative velocity vector has a longitudinal direction as shown and will therefore polish the specimen longitudinally even though the belt has a circumferential velocity component. This longitudinal polish is, of course, desired for bending fatigue specimens.

This same arrangement can be used to polish in essentially a circumferential direction merely by reversing motor *M*. If the specimen rotation is reversed so that its surface velocity is V'_S downward, then the velocity of the belt *relative* to the specimen is the vector difference between V'_S and V_B, which is V'_R and is essentially circumferential.

In polishing a specimen, progressively finer sanding belts are used and the direction of motor *M* is reversed every time the belt grit size is reduced. The alternating circumferential-longitudinal polish produces new polishing scratches which are easily distinguishable from the old ones since they are essentially perpendicular to each other. Thus the amount of polishing required at each stage is easily determined. Grade 500 grit sanding belts are generally used for the final polish, which is of course done in a longitudinal direction.

THE IMPORTANT TEST VARIABLES AND THEIR CONTROL

The inconsistency and in many cases the contradictions existing in present day literature on damping are to large extent due to lack of control of the important test variables. In fact, in many cases a state of unawareness exists concerning such important variables as stress, stress history, rest, and frequency. It is therefore desirable to list the important variables in damping and elasticity research and indicate how they were handled in this work.

Variables Not Investigated But Fixed as Indicated:

(*a*) *The Material and Its Thermal Treatment.*—Electric melted SAE 1020 hot-rolled mild steel was used in this program. Further details on this material and its static properties are given elsewhere (1).

Specimens finished in the contour-block polisher during the early stage of this program were generally stress relieved 100 min at 890 F in a helium atmosphere. This was done to minimize the scatter observed in damping properties, which are highly sensitive to the cold-worked or buffed condition of the surface layer caused by the machining and polishing process. Data procured with specimens heat treated as indicated above are identified by the term "stress relieved."

Specimens finished in the controlled-pressure polisher were not stress relieved unless otherwise indicated.

(*b*) *Testing Temperature.*—Tests were conducted at 80 ± 10 F, air blast cooling being used as required during high stress, high speed runs.

(*c*) *Type of Stress and Stress Ratio.*—Rotating-bending stress is imposed by the rotating cantiliver-beam test. This results in reversed stress (from a positive stress to an equal negative stress).

(*d*) *Stress Distribution in Test Specimens.*—The hollow, tapered specimen used has a uniform longitudinal stress distribution and a radial stress distribution in which the stress on the inside surface is approximately 10 per cent smaller than that at the outside surface. Furthermore, being a rotating-bending test, all outer fibers are exposed to the maximum stress (neutral bending axis of specimen rotates relative to specimen). Consequently, practically the entire volume of the test section is exposed to stress ranging from 90 to 100 per cent of maximum.

Important Test Variables Investigated:

(*a*) *Magnitude of Reversed Stress.*—Various stresses to approximately 150 per cent of the fatigue limit were studied. Each specimen was generally tested at a constant stress.

(*b*) *Frequency of Cyclic Stress.*—Considering the simplified case of sinusoidal stress variation (no harmonics) two frequency variables should be considered: (*1*) the frequency of the cyclic stress imposed prior to measurement (stress history frequency), and

(*2*) the frequency of the cyclic stress at the time readings are taken.

It was determined in earlier work that the frequency of the cyclic stress making up the stress history does not appear to be significant. Apparently, only the total number of cycles and the corresponding stress magnitude, irrespective of frequency, are the determining factors in defining stress history. Frequencies in the range from 20 to 10,000 cpm were generally used to build up a stress in the work reported herein.

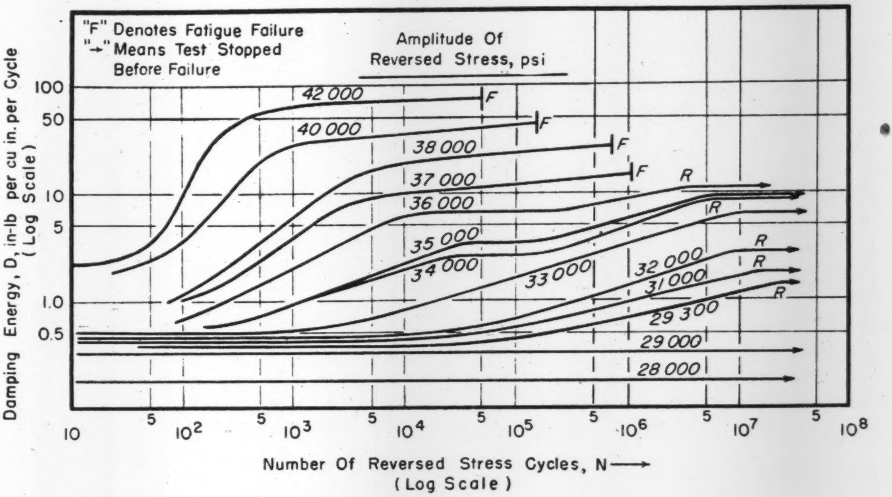

FIG. 6.—*D-N-S* Diagram Showing Effect of Sustained Reversed Stress of Several Magnitudes on Damping Energy of Stress Relieved-Mild Steel.

Frequency during reading is, however, an important test variable as discussed in the section on "Effect of Frequency of Cyclic Stress and Strain Rate," and is therefore carefully controlled in this work. Unless otherwise stated all damping and elasticity data were procured at 20 cpm.

(*c*) *Stress History.*—Stress history, one of the important parameters under study in this work, may be defined in the following general terms:

(*1*) the number of previous stress cycles and the corresponding stress amplitude, and

(*2*) time at rest at zero stress after a stress history of type (*1*) above.

The inclusion of all the combinations of variables suggested by the above general terms is of course a tremendous task. Thus, stress history used in this work was simplified to include only reversed *constant magnitude* bending stress, unless otherwise stated. Furthermore, for additional simplification, damping and dynamic modulus were measured at the stress at which the history was imposed, unless otherwise stated.

Although the study of the effect of rest from cyclic stress was not planned at this time, nevertheless exploratory work in this field was undertaken as discussed in the section on "Effect of Rest at Zero Stress."

EFFECT OF STRESS HISTORY

As indicated under "Test Equipment," it is possible to continuously measure damping and dynamic modulus in the rotating cantilever machine during the course of a reversed-bending fatigue test. Figure 6, which includes some of earlier data (1) and new low stress data, shows the variation in damping energy during a reversed constant-bending stress fatigue test. The uppermost curve, for example, shows that at the above-fatigue-limit stress of ±42,000 psi the virgin specimen (or more accurately a specimen with only ten cycles of stress) has a damping energy of 2 in-lb per cu in. per cycle, after 100 cycles of ±42,000 psi fatigue stress the damping is 10, after 1000 cycles the damping is 70, and finally after 50,000 cycles the damping attains an ultimate value of 80 as the specimen fails in fatigue. The below-fatigue-limit stress of ±32,000 psi may be considered as a second example. Here the damping does not increase significantly until after 10,000 cycles of stress, then changes from approximately 0.5 to 3.0 at 10^7 cycles (point *R*), beyond which the damping does not change under additional reversed stress. This point, *R*, at which the damping becomes stable with respect to additional cyclic stress shall be defined as the "stabilized damping point." At the relatively low stresses of 28,000 and 29,000 psi the damping capacity is not affected by sustained reversed stress; in one sense the "stabilized damping point" occurs at 1 cycle. For the particular series of specimens diagrammed in Fig. 6, it may be observed that either a specimen fails in fatigue (see points *F*), or it attains a condition of stabilized damping (see points *R*) after which fatigue failure appears to be extremely unlikely.

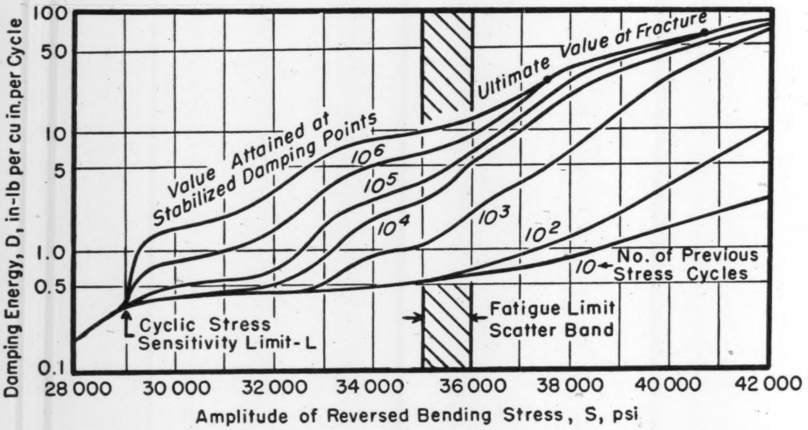

FIG. 7.—*D-S-N* Diagram Showing Effect of Reversed Stress Magnitude and Stress History on Damping Energy of Stress-Relieved Steel.

The significance of the stabilized damping point and the generality of its occurrence in other materials is not completely clear at present. Early investigations have implied that stabilized damping is attained in a fatigue test. Bairstow (3) reported that for axle steel (55,000 psi yield strength and 85,000 psi tensile strength) the "cyclical permanent set" (which corresponds to the width of the hysteresis loop) attains a definite constant value after approximately 10,000 stress cycles at stresses both below and above the fatigue limit. However, in Bairstow's experiments, the stress on each specimen was increased stepwise (virgin specimens not used at each different stress level). Furthermore, relatively few cycles of stress were used and although the linear plots may indicate impending stabilization at 10,000 cycles logarithmic plots such as Fig. 6 may indicate an entirely different picture. Gough (4) has observed a similar stabilized damping behavior during cyclic stress tests in Armco iron, which he identified by the controversial term "elastic hysteresis." However, none of the early investigators apparently covered a wide range of stress on virgin specimens subjected to a large number of cycles. The concept of stabilized damping is discussed in greater detail in later sections.

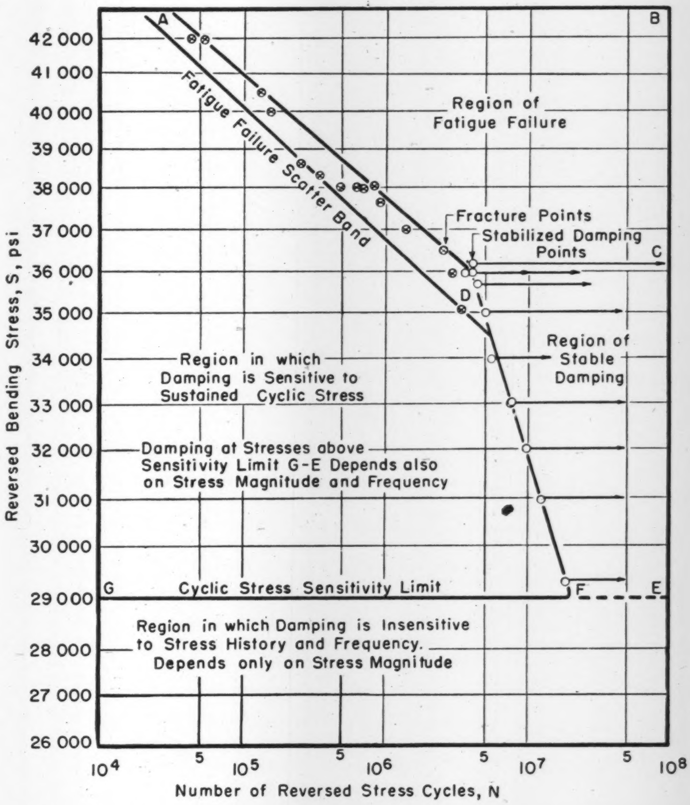

FIG. 8.—*S-N-N* Fatigue Diagram Extended to Include Stabilized Damping Line for Stress-Relieved Mild Steel.

The *D-N-S* curves shown in Fig. 6 are replotted in Fig. 7 as *D-S-N* curves in order to emphasize the concept of a "cyclic stress sensitivity limit," *L*. This limit, defined (1) as the cyclic stress below which damping is independent of stress history, is approximately 29,000 psi for the stress-relieved mild steel under study. Each curve on this diagram represents the damping after the number of reversed *constant* bending stress cycles indicated by the figures, the damping measurements being made at the same stress (see abscissa) as that used in the stress history. It is quite apparent from this diagram that above the sensitivity limit, *L*, stress history influences damping to a larger extent in most cases than does magnitude of stress in the range covered. For example, the highest damping attainable in a virgin specimen at the fatigue limit is approximately 0.6 in-lb per cu in. per cycle, whereas after a stress history which brings the specimen to the stabilized damping point a value 12 (twenty times the virgin value) can be attained.

ing is not only dependent on stress magnitude and frequency, but also greatly dependent on stress history.

3. Stable damping region *C-D-F-E*, in which damping during constant reversed stress depends only on stress magnitude and frequency and does not change during sustained cyclic stress.

4. The low stress region below the cyclic stress sensitivity limit *G-F-E*, in which damping is insensitive to stress history and frequency and depends only on stress magnitude.

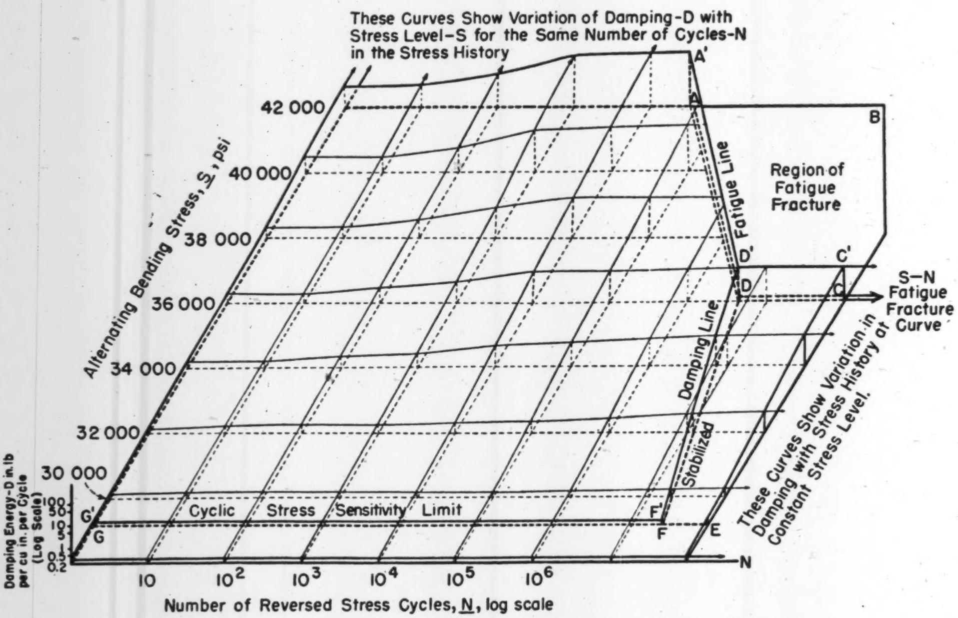
These Curves Show Variation of Damping-D with Stress Level-S for the Same Number of Cycles-N in the Stress History
A'
A
B
Region of Fatigue Fracture
Fatigue Line
D'
C'
D
C
S-N Fatigue Fracture Curve
Damping Line
Stabilized
These Curves Show Variation in Damping with Stress History at Constant Stress Level.
Alternating Bending Stress, S, psi
42 000
40 000
38 000
36 000
34 000
32 000
30 000
Cyclic Stress Sensitivity Limit
G'
G
F'
F
E
N
Damping Energy-D in.lb per cu in. per Cycle (Log Scale)
100
50
10
5
1
0.5
0.2
10
10^2
10^3
10^4
10^5
10^6
Number of Reversed Stress Cycles, N, log scale

FIG. 9.—*S-N-D* Diagram Indicating Relationships Among Damping Energy, Stress Magnitudes, Number of Reversed Stress Cycles, and Fatigue Failure for Stress-Relieved Mild Steel.

Observations concerning the existence of stabilized damping points *R* and a cyclic stress sensitivity limit *L* discussed above have been included in the extended *S-N* diagram shown in Fig. 8. This *S-N-N* diagram indicates stress *versus* (*a*) number of cycles to fatigue failure and (*b*) number of cycles required for stabilizing the damping. There are four distinct regions in this diagram:

1. Fatigue failure region *A-D-C-B*.
2. Region *A-D-F-G*, in which damp-

Referring to Fig. 8, it may be seen that the stabilized damping points fall along straight line *D-F* with smaller scatter than that displayed by the fracture points. Although it is realized that considerably more data is required before scatter, distribution, and statistical factors can be thoroughly analyzed, a preliminary discussion of difference in scatter is given below.

According to Orowan (5) and others, fatigue failure in a specimen is caused by the cumulative effects occurring in structurally inhomogeneous regions which display highly localized stress. Stresses are highly concentrated in these regions when the specimen is externally loaded. The peak stresses in these regions may be relaxed or partly relieved by the elastic surroundings under certain conditions of localized plastic deformations. It may generally be assumed that there are a great number of structurally inhomogeneous regions randomly distributed in a fine-grained polycrystalline specimen. The resultant plastic deformations in these regions manifest themselves as hysteresis loss during cyclic loading. The damping at any stress level, and in particular at stabilized damping points, is determined by the statistical average of the products of the many localized plastic deformations and their corresponding stress. Therefore, the location of the stabilized damping points along line *D-F* should be characteristic of their respective stress levels within the limit of experimental error.

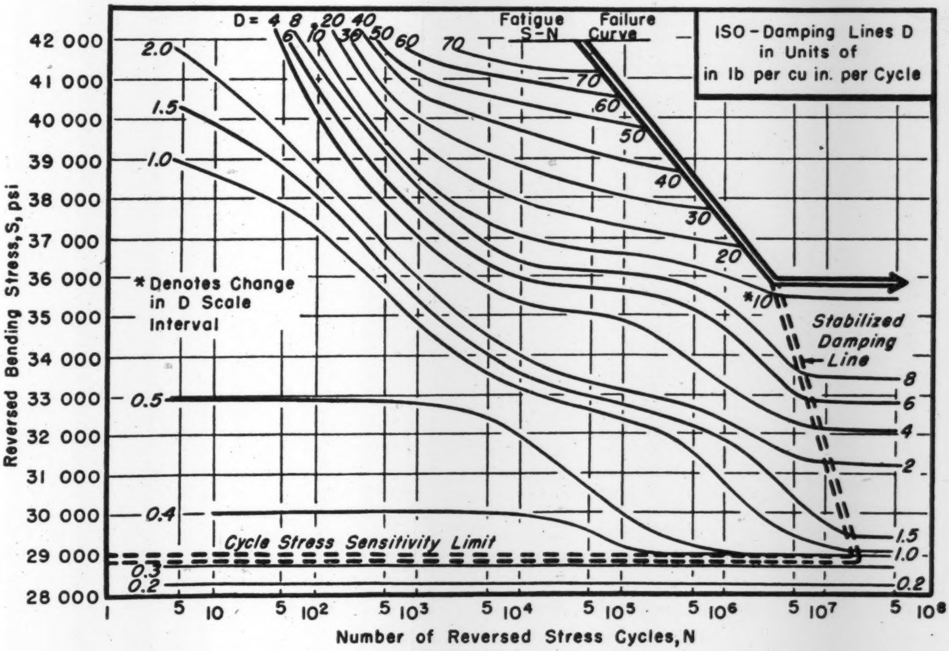

FIG. 10.—*S-N-D* Diagram Relating Stress, Number of Cycles, and Iso-Damping Lines for Stress-Relieved Mild Steel.

The number of cycles at which fatigue fracture occurs depends not only on the localized stress in the structurally inhomogeneous regions and the accumulative effects as explained above but also on the mechanism of crack formation and propagation. Microcracks in a fatigue specimen tested above the fatigue limit are probably formed only at the more severely inhomogeneous regions in the specimen. Furthermore, the propagation of these microcracks into fatigue microcracks, and their progressive growth to macrocracks, is further complicated by other factors, such as the size and orientation of grains, type of

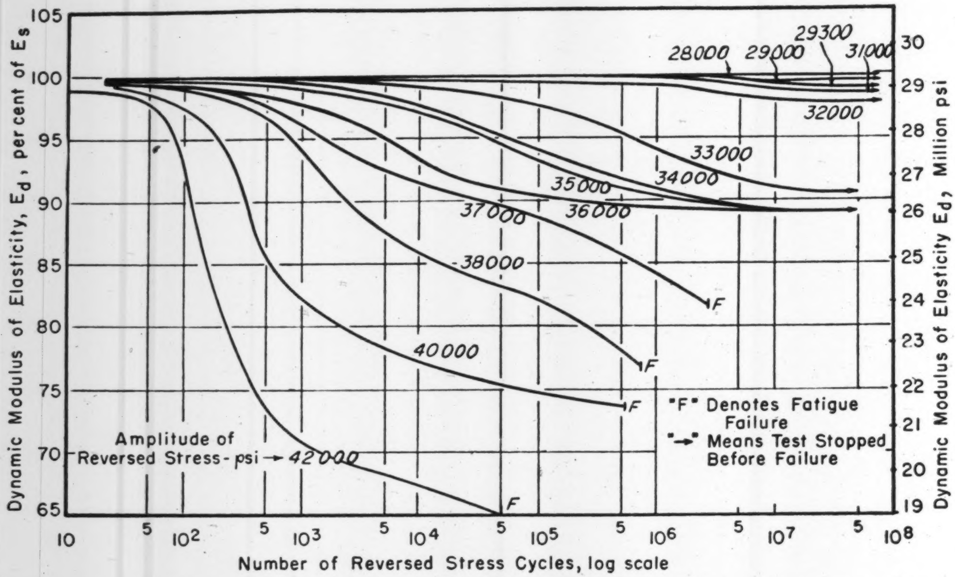

FIG. 11.—E_d-N-S Diagram Showing Effect of Sustained Reversed Stress of Several Magnitudes on Dynamic Modulus of Elasticity for Stress-Relieved Mild Steel.

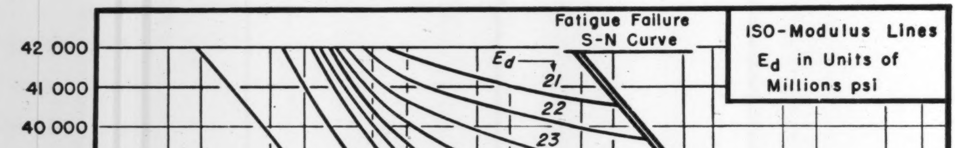

FIG. 12.—S-N-E_d Curves Relating Stress, Number of Cycles, and Iso-Modulus Lines for Stress-Relieved Mild Steel.

loading, and stress gradients and distribution. For these reasons the greater scatter in the fracture points as compared to the stabilized damping points appears logical.

The scatter in fracture point data is generally larger in the vicinity of fatigue limit than elsewhere. Thus, the establishment of a fatigue limit within reasonable accuracy generally requires a sufficiently large sample to permit use of statistical methods. However, statistical methods are extremely tedious and time consuming. Consequently, there is considerable need for a method of determining the fatigue limit which would reduce the number of specimens required for a reliable determination. The approach suggested by Fig. 8, in which the knee of the *S-N* curve may be determined from the intersection of the fatigue failure line and the stabilized damping line, shall be explored further in future work. Since all materials probably do not display stabilized damping points, this method may have limited applicability.

The damping and fatigue data shown in Fig. 7 and 8 are presented in the three-dimensional *S-N-D* diagram of Fig. 9. In this diagram the damping is represented as the height to the surface above the *S-N* base plane. The slope of this surface indicates the rate of change of damping, either with respect to changing stress magnitude, *S*, or changing stress history, *N*. This surface ends abruptly, of course, at the *S-N* fatigue failure abyss defined by fracture line *A'-D'*. In the below-fatigue-limit tests damping becomes constant beyond the stabilized damping line *D'-F'*, and the surface becomes a surface having zero slope in the horizontal *N* direction but one that goes uphill with increasing stress. Below the cyclic stress sensitivity limit *G'-F'* the surface is also level in the horizontal *N* direction and it slopes downward towards zero stress.

The *S-N-D* surface of Fig. 9 may be shown in a form involving use of iso-damping contour lines, or lines of equal damping, as illustrated in Fig. 10. These iso-damping lines, which are similar in nature to contour lines on a map for showing points of equal elevation, indicate trends and rapidity of change (uphill and downhill) in damping. The horizontal contour lines to the right of the stabilized damping line and below the cyclic stress sensitivity limit indicate the insensitivity of damping to additional cyclic stress in these regions. This diagram also permits quantitative determination of damping as a function of stress and number of cycles and consequently can be considered useful as a design chart.

The data on dynamic modulus of elasticity, E_d, also procured during these same tests, may be treated in a manner similar to that shown in Figs. 6 to 10 for damping energy. However, for conciseness only the E_d-*N*-*S* and *S*-*N*-E_d diagrams for dynamic modulus, Figs. 11 and 12, respectively, are included in this paper. The rather large decrease in dynamic modulus under sustained reversed stress is significant, as much as 11 per cent at stresses below the fatigue limit and as much as 35 per cent at a stress 20 per cent above the fatigue limit. The *S*-*N*-E_d diagram has many characteristics similar to the *S-N-D* diagram. The decrease in modulus is roughly reciprocally related to the increase in damping and stability regions are also similar. Reasons for these relationships between damping and dynamic modulus have been discussed previously (1).

All the specimens diagrammed in Figs. 6 to 12, were prepared in the contour-block polisher and stress relieved at 890 F for 100 min. For comparison purposes a series of six specimens were prepared in the controlled-pressure belt polisher. These specimens were not stress relieved after final polishing since tests indicate that the cold worked or buffed condition

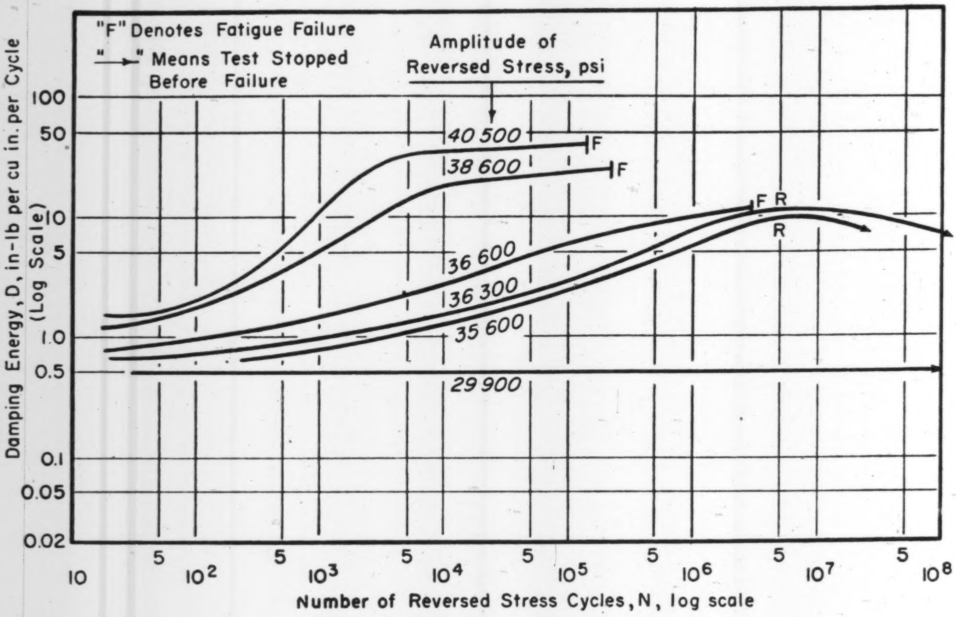

FIG. 13.—*D-N-S* Diagram Showing Effect of Sustained Cyclic Stress of Several Magnitudes on Damping Energy for Mild Steel.

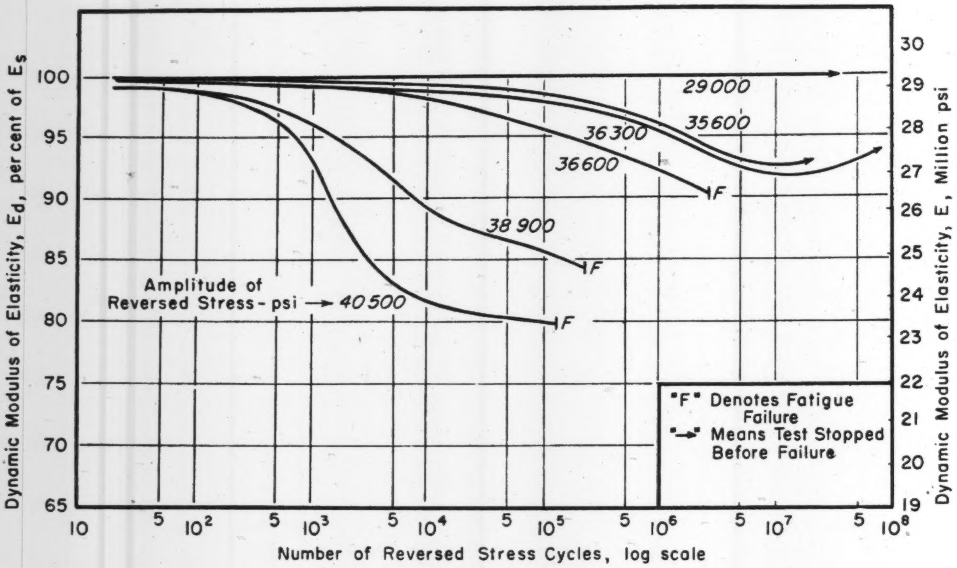

FIG. 14.—*E_d-N-S* Diagram Showing Effect of Sustained Reversed Stress of Several Magnitudes on Dynamic Modulus of Elasticity for Mild Steel.

of the surface polished under controlled pressure is probably significantly less serious than that polished with the contour block. D-N-S and E_d-N-S data for these specimens, diagrammed in Figs. 13 and 14, indicate the following differences, when compared to Figs. 6 and 11 for stress-relieved specimens:

1. Their damping is somewhat lower particularly after some stress history, than that of the stress-relieved specimens.

2. Their damping curves for stress near ±36,000 psi display fewer discontinuities.

3. These specimens do not, like the stress relieved specimens, appear to attain the condition of constant damping in the stabilized damping region. Rather, there is a small but probably significant decrease in damping in what is the stabilized damping region for stress relieved specimens. It is entirely possible, of course, that if the stress relieved specimens were carried sufficiently far beyond the stabilized damping line, they too would display a downward trend. However, no such trend was observed up to 10^8 cycles in one test below the fatigue limit.

In view of the above observations, it may be necessary to redefine the stabilized damping point in future work to permit inclusion of more materials.

The explanation of the above differences in damping from the physics of metals viewpoint and the determination of the degree of surface cold-working resulting from the two polishing methods are beyond the scope of this paper.

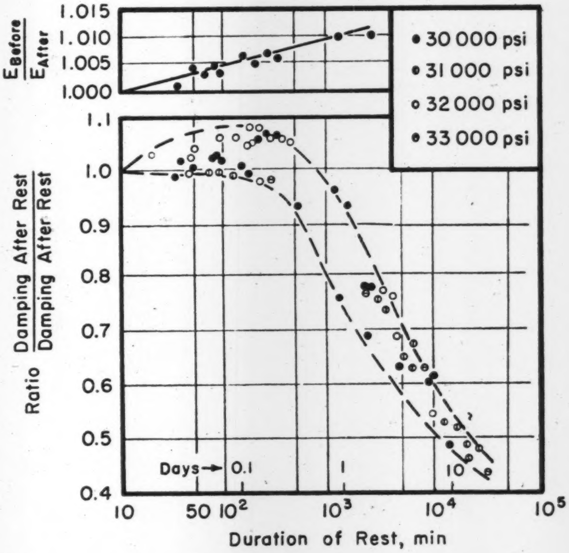

FIG. 15.—Effect of Rest on Damping and Dynamic Modulus of Elasticity for Mild Steel in the Stabilized Damping Region.

EFFECT OF REST AT ZERO STRESS

Although the study of the effect of rest from cyclic stress was not to be included in this program, nevertheless, exploratory tests of this type were undertaken. One purpose of these tests was to indicate the maximum time at rest allowable after a stress history without changing damping or dynamic modulus. Such information is necessary in planning testing techniques so that rest between readings does not significantly affect damping and modulus while studying other variables.

In order to procure data on the effect of rest a series of specimens was subjected to sustained reversed constant stress of various magnitudes until stabilized damping was attained. The specimens were then allowed to rest at zero stress and periodic measurements on damping and modulus were made at the original stress magnitude in order to study the change in damping and modulus during the rest period. The approximately 20 cycles of stress required to make these measurements have little effect on damping.

In most cases, there is a very small increase in damping during the first part of the rest followed by a pronounced decrease. This is shown in Fig. 15, in which the ratio of damping after rest to damping before rest is plotted as a function of time of rest. Similar data is also given for dynamic modulus.

It may be seen from this figure that for the range of conditions investigated, rest up to a few hours does not seriously affect the damping and modulus values. Since in most cases only a few minutes of rest generally occur between readings in the standard test, it may be concluded that rest effects were controlled in this work.

DYNAMIC STRESS-STRAIN CHARACTERISTICS

In order to better understand the nature and physical causes of the change in damping and dynamic modulus during a fatigue test, a series of tests were undertaken to determine the dynamic stress-strain characteristics of mild steel. This work was planned also to help clarify the relationship between damping and dynamic modulus.

Before presenting and discussing the dynamic stress-strain data procured, it is desirable first to discuss the significance, in terms of stress-strain curves, of the damping and elasticity measurements made with the rotating cantilever equipment. The stress-strain relationships of a material under reversed stress may be represented by exaggerated hysteresis loop *A-B-C-D-A* in Fig. 16. The area of this loop, which may be determined directly from the horizontal traversal *H* in the rotating cantilever-beam test, is a measure of the damping energy of the material. Maximum strain, ϵ_{max}, designated by *T*, is determined from the vertical deflection *V* in the rotating-beam test and may be used to determine secant modulus of elasticity at *A* (proportional to the slope of line *O-A*). This secant modulus during cyclic stress is called the dynamic modulus of elasticity. At

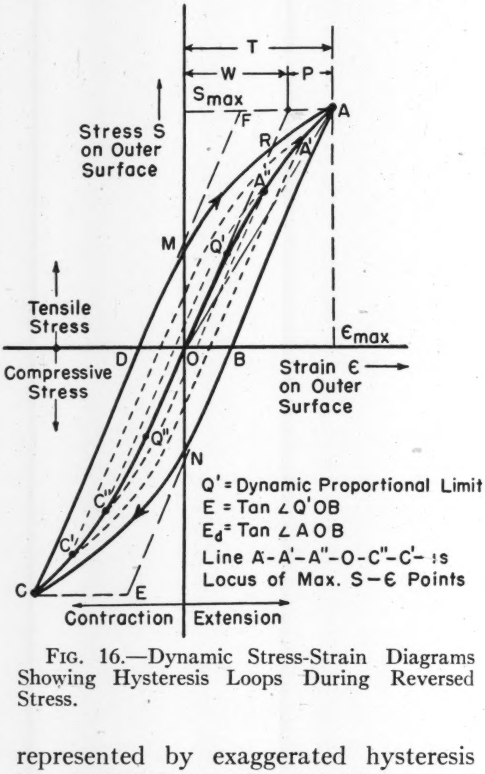
T
W
P
Smax
F
A
R
A'
A''
Stress S on Outer Surface
M
Q'
Tensile Stress
εmax
D
O
B
Compressive Stress
Strain ε on Outer Surface
Q''
N
C''
C'
C
E
Contraction
Extension
Q' = Dynamic Proportional Limit
E = Tan ∠ Q'OB
Ed = Tan ∠ AOB
Line A-A'-A''-O-C''-C'- is Locus of Max. S-ε Points

FIG. 16.—Dynamic Stress-Strain Diagrams Showing Hysteresis Loops During Reversed Stress.

lower stress levels A' and A'', smaller hysteresis loops generally will be developed and higher dynamic moduli of elasticity (proportional to the slope of lines OA' and OA'', respectively) generally will be displayed.

The best method of studying the dynamic stress-strain behavior is to procure actual hysteresis loop records of the instantaneous stress-strain relationships. However, suitably simple and reliable equipment to do so has not yet been developed. Therefore, at present, the dynamic stress-strain characteristics must be inferred from rotating bending *H* and *V* measurements as explained below.

In the series of tests to be discussed vertical deflection readings, *V*, were taken continuously and rapidly (so that stress history factors do not become significant) as the magnitude of cyclic stress was gradually increased. These *V* readings determine curve *O-Q'-A''-A'-A* (or *O-Q''-C''-C'-C*), hereafter called the "dynamic stress-strain curve." This curve does not, of course, represent the true instantaneous stress-strain relationship, which is *A-B-C-D-A*, but is rather the locus of maximum stress-strain points Q', A'', A', *and* A during increasing load. Point Q' at which the dynamic stress-strain curve deviates from a straight line may be called the "dynamic proportional limit." It is, of course, recognized that a true dynamic proportional limit probably does not exist since inelasticity is present at all stresses, and the apparent values measured are a function of instrument sensitivity. However, the observable deviation from stress-strain linearity does increase rapidly under cyclic loading beyond a certain point, just as it does under static loading. Thus the determination of the apparent limits is generally not too difficult. Although the proportional limits referred to in the following discussion are not the true values, but rather the apparent limits (instru-

Specimen	PA 112		PA 196			PA 108		PA 244				
Maximum Reversed Stress	29 900		31 100			35 600		38 000 psi				
Run Designation	A	B	C	D	E	F	G	H	I	J	K	L
Prior Cycles at Max Stress	20	30×10^6	20	1400	14×10^6	20	15×10^6	20	1640	4500	5×10^4	10^5 +
Tangent Modulus, 10^6 psi	29.4	29.4	29.4	29.4	29.4	29.4	29.4	29.4	29.2	28.8	28.6	28.5
Plastic Strain P, 10^4 in. per in.	0	0	0	0.2	0.6	0	1.2	0.2	1.2	2.2	2.5	2.8
Static Prop Limit Q - psi	> 29 900		>31 000	25 000	19 000	> 35 600	23 000	> 38 000	19 000	17 000	16 000	16 000
Dynamic Prop Limit Q'- psi	> 29 900		>31 000	27 000	21 000	> 35 600	25 000	35 000	23 000	21 000	19 000	18 000

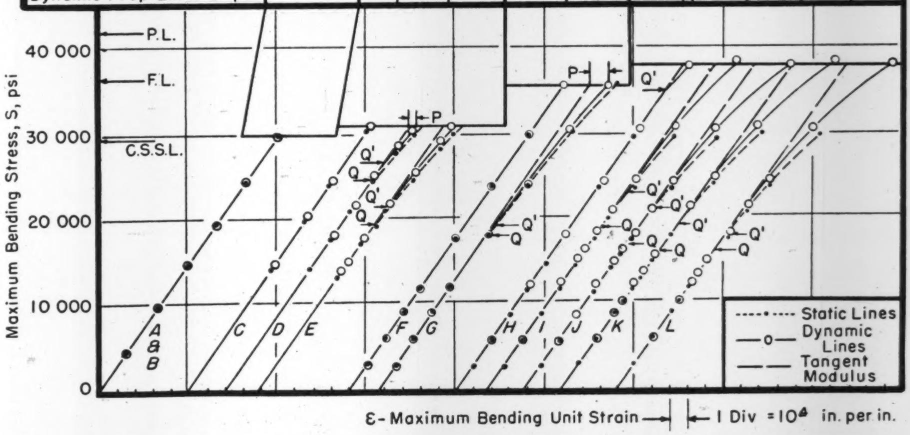

FIG. 17.—Effect of Stress History on Static and Dynamic Stress-Strain Curves for Mild Steel.

ment sensitivity at limit approximately 0.1 per cent), the word apparent is omitted for conciseness.

The results of tests illustrating the dependence on stress history of the static proportional limit Q, the dynamic proportional limit Q', and other dynamic stress-strain characteristics are diagrammed in Fig. 17. Referring first to specimen PA112, static load-deflection data (solid points) were first procured on the virgin specimen up to 29,900 psi, and then dynamic load-deflection data (hollow points) were determined at 20 rpm. Both the static and dynamic stress-strain curves coincide (curve A). Thirty million cycles at ±29,900 were then imposed and again static and dynamic stress-strain characteristics were procured and plotted (curve B). All data at 29,900, both before and after the stress history, coincide with each other. This is to be expected since at no time did the stress exceed the cyclic stress sensitivity limit.[4]

The tests on the other three specimens diagrammed, specimens PA196, PA108, and PA244, were conducted above the cyclic stress sensitivity limit. In the first two cases, the virgin specimens were elastic (no plastic strain component P) up to the test stress. Specimen PA244, tested above the fatigue limit, displayed a dynamic proportional limit Q' of approximately 35,000 psi. In all three cases sustained cyclic stress progressively and pronouncedly reduced both the static and dynamic proportional limit, Q and Q', the former more than the latter.

The change in static proportional limit demonstrated in Fig. 17 for the high stress level tests was first observed by Bauschinger (6) and later by other investigators. Bauschinger designated the static proportional limit of the virgin specimen the "primitive elastic limit" (approximately 41,000 psi in bending for the mild steel under investigation) and he called the static proportional limit after cyclic loading the "natural proportional limit," here defined as Q.

Even though sustained reversed stress between the cyclic stress sensitivity and the fatigue limit (specimens 196 and 108) causes pronounced changes in several properties as discussed above, stress history does not affect either the static or dynamic tangent modulus at zero stress. However, in the above-fatigue-limit test at 38,000 psi the tangent modulus does not remain constant; at the half life of the specimen the tangent modulus at zero stress was 3 per cent lower than in the virgin specimen. Although a great deal of additional data is necessary before this observation can be generalized with regard to all above-fatigue-limit tests, nevertheless several such tests conducted on mild steel to date indicate such a reduction in tangent modulus.

The change in tangent modulus during a stress history above the fatigue limit, as discussed above, does not confirm Bairstow's work (3), which indicates that the "initial elastic line" remains unchanged through all stress ranges and stress histories. However, Bairstow used only a few hundred stress cycles in tests above the fatigue limit, and this is generally insufficient to reveal the trends.

Gough (4) and others, in an attempt to develop an accelerated fatigue limit determination, have tried to associate what has been defined here as the dynamic proportional limit Q' with the fatigue limit. This limit was determined on one specimen under continuously increasing stress. In view of the sensitivity of the test material to stress history, it might appear to be difficult to avoid the stress history variable as a distorting influence in determining the dynamic proportional limit of the virgin material. However, referring to Fig. 11 it may be seen that below the fatigue limit stress history does not exert an immediate influence; it generally requires a few hundred cycles to cause a significant change. Thus, if measurements are made very rapidly, say in less than 50 cycles per reading, the stress history influence is generally insignificant. Dynamic stress-strain curves procured at 20 rpm and rapidly enough to make stress history insignificant are shown in Fig. 17, Run H, and Fig. 18. In both cases, the dynamic proportional limit is approximately 35,000, which is within the fatigue limit scatter band. As a matter of interest, damping data procured during the same test is also shown in Fig. 18.

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[4] According to Fig. 7, the cyclic stress sensitivity limit is 29,000 psi for stress-relieved specimens. The cyclic stress sensitivity limit of this series of specimens, finished in the controlled pressure polisher and not stress-relieved, appears to be above 29,900 psi (see Figs. 13 and 14). However, considerably more data is required to establish this limit accurately since the scatter range is rather large.

Gough (4) has found that the dynamic proportional limit is a good indication of the fatigue limit for a large number of ferrous materials, with this limit particularly abrupt and sharply defined for ductile ferrous metals. He found this method less reliable for non-ferrous metals, and in case of some aluminum alloys quite misleading.

Additional work on the association of the dynamic proportional limit with the fatigue limit is being planned for future programs. Materials without a well defined fatigue limit present a particularly interesting case.

EFFECT OF FREQUENCY OF CYCLIC STRESS AND STRAIN RATE

Some of the early investigators assumed that damping of metals was viscous in nature and therefore increased with frequency. The incorrectness of this assumption has been proved by many investigators. In Rowett's work (7) on steel in torsion and Kimball's study (8) on several metals in bending, damping was observed to be independent of frequency over a rather, wide range. In cases where frequency sensitivity has been reported (9) the stresses have been extremely low[5] or frequency has been very high. Very little data dealing with the effect of frequency of cyclic stress on damping and dynamic modulus in the engineering range of stress have been published. What few data have been presented have indicated that damping is independent of frequency (10) at engineering stresses.

It is shown in this paper that the above mentioned belief that the damping of mild steel is independent of frequency is true only in the limited range of stress below the dynamic proportional limit. Data presented below show that above the dynamic proportional limit, where the plastic strain component becomes significant, damping and dynamic modulus are dependent on frequency.

In the first report (1) on this work, frequency effects for mild steel at stresses near the fatigue limit were summarized as follows for the range from 20 to 1800 cycles per minute:

1. Increased frequency at stresses near the fatigue limit causes a decrease in damping and an increase in dynamic modulus. The instantaneous values are therefore frequency sensitive.

2. In spite of this frequency sensitivity of the instantaneous values, the frequency of the cyclic stress during a previous stress history does not greatly affect the change in damping (or modulus) caused by a given number of stress cycles. Stated differently, the rate of change of damping per cycle is relatively insensitive to frequency.

In the current work it was decided to determine the effect of frequency over a range from 0.05 to 10,000 cycles per

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[5] At very low stress (say a few pounds per square inch the major causes of damping, such as thermal currents, grain boundaries, etc., are frequency sensitive. However, these factors contribute only a very small part of the total damping displayed at engineering stress levels, and therefore the total damping at high stresses may practically lose its frequency sensitivity.

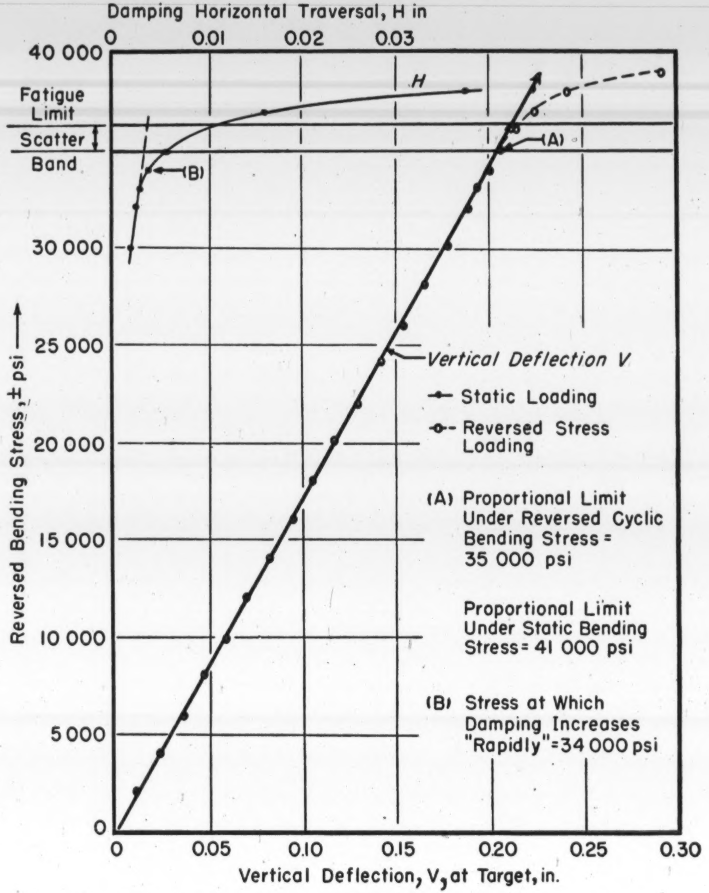

FIG. 18.—Relationships Between Fatigue Limit and Dynamic Proportional Limit and Damping Phoperties for Mild Steel.

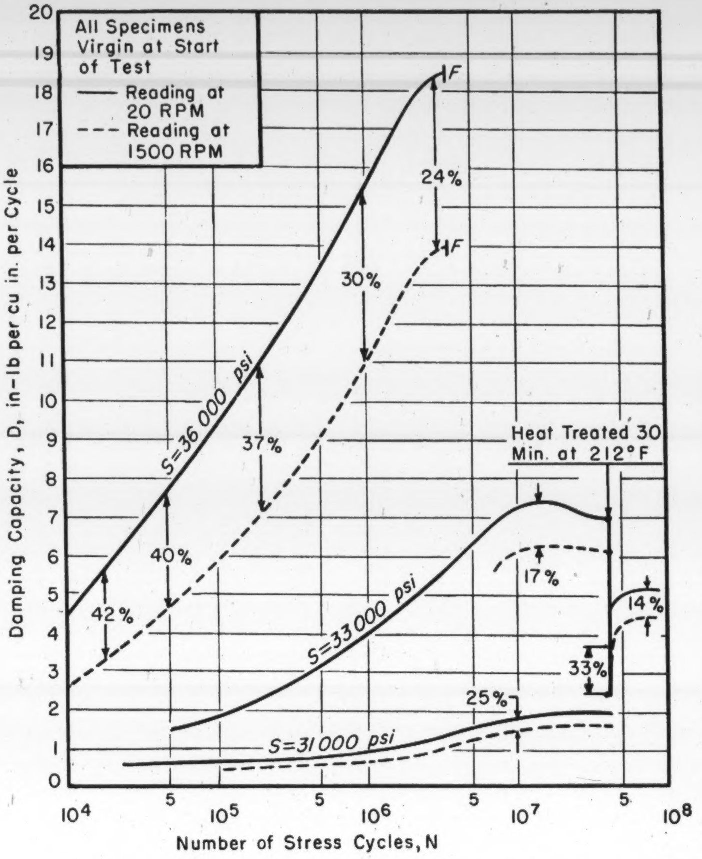

FIG. 19.—Effect of Frequency on Damping of Mild Steel at Various Stresses and Stress Histories.

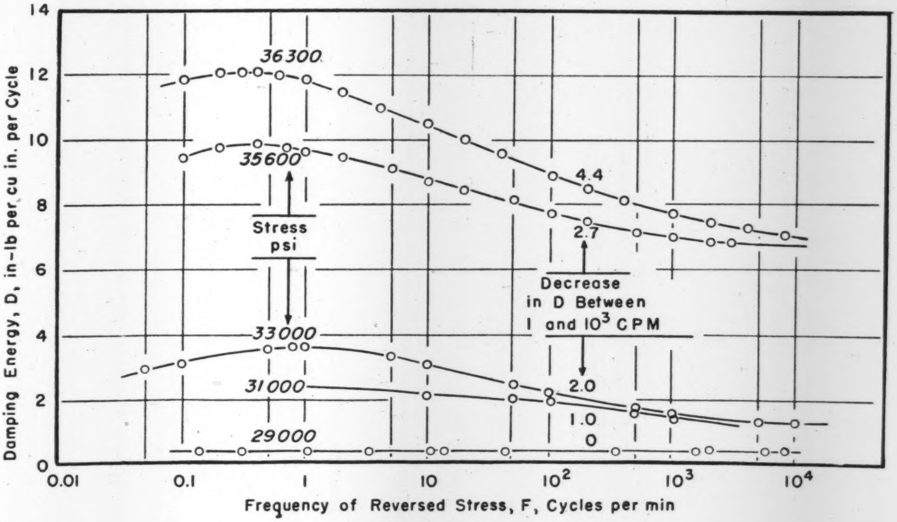

FIG. 20.—*D-F-S* Diagram Showing Effect of Frequency of Reversed Stress on Damping Energy at Several Stress Magnitudes for Mild Steel in the Stabilized Damping Region.

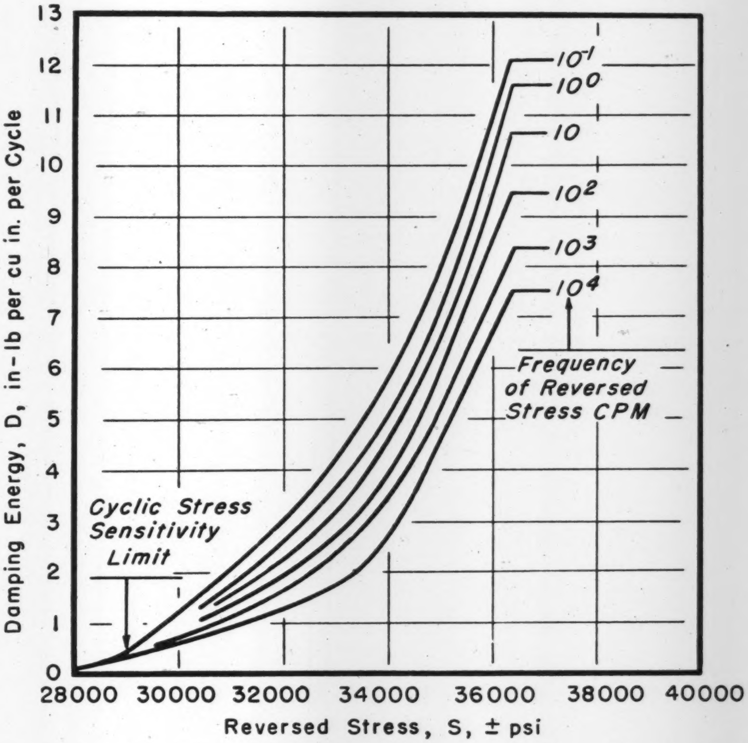

FIG. 21.—Effect of Frequency of Cyclic Stress on Damping-Stress Relationship for Mild Steel in the Stabilized Damping Region.

minute on the instantaneous values of damping and modulus (data of type 1 above). However, before doing so it was necessary to undertake preliminary work to assure separation of stress history effects from the frequency effects. Part of the data procured in this preliminary work are shown in Fig. 19.

These preliminary tests show that frequency sensitivity depends not only on stress level but also on stress history. The test at 36,000 psi for example, shows (Fig. 19) that both the absolute and percentage change in damping caused by a given frequency change varies during the sustained cyclic stress test at constant stress level. It is thus apparent that it is rather difficult to procure frequency sensitivity data on virgin specimens as a function of stress without involving stress history effects.

It is also difficult to procure frequency sensitivity data at high frequency on a virgin specimen without radically changing the stress-history factor. For example, referring to Fig. 6, if only 2 min were required to procure the 38,000 psi damping and elasticity data at a frequency of 10,000 rpm, then the 20,000 cycles imposed during the reading process would change the damping in the virgin specimens from approximately 0.8 to approximately 20. Needless to say, such large changes due to stress history would make it difficult to separate frequency effects.

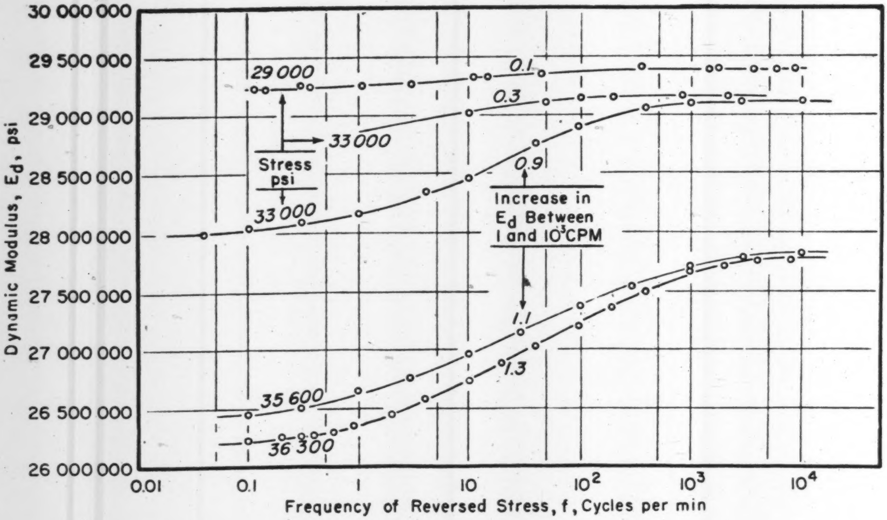

FIG. 22.—E_d-F-S Diagram Showing Effect of Frequency of Reversed Stress on the Dynamic Modulus at Several Stress Magnitudes for Mild Steel in the Stabilized Damping Region.

In order to remove these stress-history difficulties, it was decided to measure frequency sensitivity in the stabilized damping region. Thus, all of the following frequency sensitivity data were procured after sufficient stress history had been imposed at reversed constant stress to bring the specimen past the stabilized damping line (see Fig. 10).

A special worm drive was incorporated in the rotating-beam dynamic testing machine so that testing speeds as low as 0.01 rpm could be used. Since the damping in high stress tests at high speed may heat the specimen significantly, air stream cooling was used at all speeds to maintain the specimens at room temperature. These and other precautions resulted in good reproducibility of the data; readings taken before and after runs, or approached from either the high or low frequency side, showed no significant scatter.

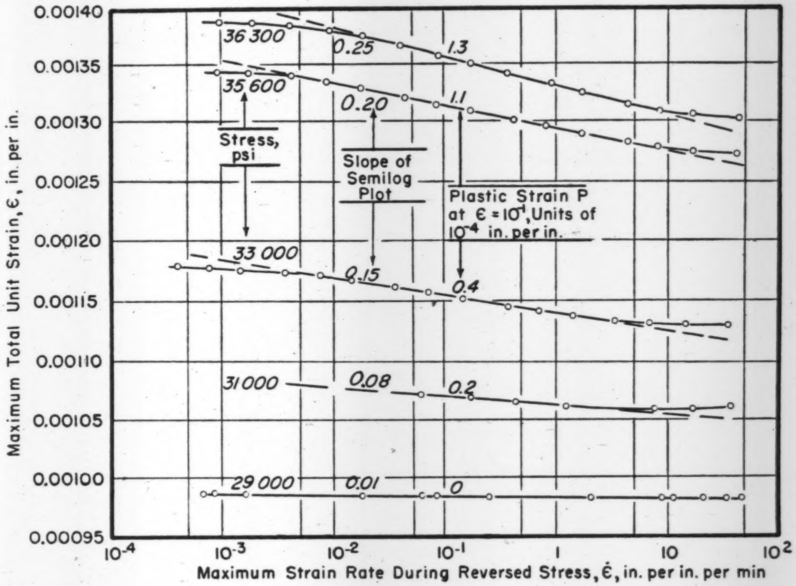

FIG. 23.—Effect of Maximum Strain Rate During Reversed Stress on Total Strain for Mild Steel Brought to Stabilized Damping Region at Stress of Measurement.

The effect of frequency on damping energy at various stress levels in the stabilized damping region is shown in Fig. 20. In each case the specimens were brought to the stabilized damping zone at the stress of measurement. Damping increases slightly with frequency from 0.05 cycles per minute, attains a peak value at approximately 0.5 cpm, and decreases significantly up to 10,000 cpm, as much as 65 per cent decrease for the test at 33,000 psi.

The relationship of frequency sensitivity to stress level is better shown in Fig. 21. At or below the cyclic stress sensitivity limit damping is independent of frequency. In general the higher the stress above this limit the greater is the frequency sensitivity. Whether the significant limit in determining frequency sensitivity is the cyclic stress sensitivity limit or the dynamic proportional limit, Q' (they both coincide for the above *constant* stress history tests), shall be discussed in a later section.

Theoretical studies of frequency patterns and relaxation spectra for metals at *very low* stress have been reported by Zener (9) who has been very successful in associating damping peaks with thermal current, solute atoms, grain boundary, and other metallurgical factors. At *high* stress, current theories (11, 12) predict an increase in damping with increased frequency, which is not in agreement with the data given above. However, the discussion of frequency effects in terms of physics of metals theory is not within the scope of this paper.

The dependency of dynamic modulus on frequency is shown in Fig. 22. As in the case of damping, dynamic modulus is also practically independent of frequency below the cyclic stress sensitivity (or the dynamic proportional limit, as shall be discussed later). Above this limit the dynamic modulus increases continuously with increasing frequency, as much as 5.7 per cent at high stress levels.

Additional frequency sensitivity work over a larger frequency range is now in progress. A rotating-beam machine with a top speed of 65,000 rpm has been constructed for this work. Aside from further extending frequency range data, damping and elasticity properties at high frequency are of considerable engineering importance since several important vibration problems occur at frequencies in the vicinity of 65,000 cpm.

The frequency sensitivity of damping and dynamic modulus shown in Figs. 20, 21, and 22 may be associated with strain rate phenomena. Since the frequency tests discussed herein were primarily exploratory in nature, and the data are very meager, a thorough analysis of this association shall not be attempted in this paper. However as an example of the development of such associations, the limited dynamic modulus data diagrammed in Fig. 22 will be interpreted in terms of strain rate as explained below.

The strain rate during the reversed stress fatigue test is, of course, not constant but sinusoidal in nature, varying between zero and maximum value as indicated below.

$$\begin{aligned} \epsilon &= \epsilon_0 \sin ft \\ \dot{\epsilon} &= \epsilon_0 f \cos ft \qquad \text{(3)} \end{aligned}$$

where:

ϵ and ϵ_0 = instantaneous unit strain and maximum unit strain, respectively, in. per in.,

$\dot{\epsilon}$ and $\dot{\epsilon}_0$ = instantaneous and maximum unit strain rate during the sinusoidal stress variation, in. per in. per min,

f = frequency of alternating strain (or stress), radians per min,

t = time, min, and

S = reversed stress, ± psi.

It may be seen from the above equation that at the instant the strain (or stress) is maximum the strain rate is zero, and at zero strain (or stress) the strain rate is a maximum having the following value:

$$\begin{aligned} \dot{\epsilon}_0 &= \epsilon_0 f \\ &= \frac{S}{E_d} f \qquad \text{(4)} \end{aligned}$$

The dynamic modulus data diagrammed in Fig. 22 may be used to compute, by means of Eq 4, the relationship between maximum strain rate and maximum strain as shown in Fig. 23. As indicated above, the maximum strain rate during a cycle occurs at zero stress, whereas the strain rate at the highest stress, (where plastic strain component P is largest) is probably the more significant. However, the average strain rate in the high stress plastic[6] region is reasonably proportional to the maximum strain rate. Therefore, even though the abscissa in Fig. 23 is maximum strain rate, rather than some other more significant measure of strain rate, the shape of the curves and trends are probably significant.

Figure 23 indicates that in general the higher the strain rate the smaller the total strain, the variation being logarithmic over a considerable portion of the range studied. Under the entirely different test condition involving unidirectional tension (load constantly increased and not repeated) over a wide range of loading speeds, Nadai (**13**) and others have found similar relationships between total strain and strain rate. The interpretation of this logarithmic relationship and the association of the various types of related data which display these patterns shall not be discussed in this paper.

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[6] The word "plastic" is not used here in the usual sense to indicate large yield since all data presented in Fig. 23 were procured below the static proportional limit of the virgin material. The phrase "plastic region" is here used to indicate stress levels where significant cyclic plastic strain P persists as shown in Fig. 17.

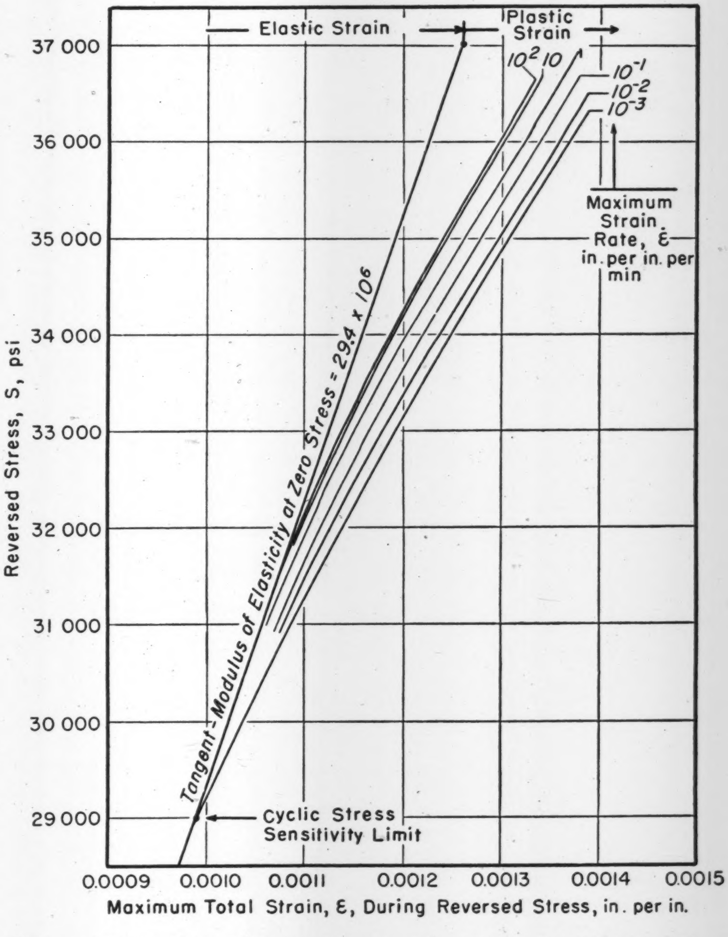

Fig. 24.—Effect of Strain Rate on Dynamic Stress-Strain Curve for Mild Steel Brought to Stabilized Damping Region at Stress of Measurement.

Still another interpretation of the frequency sensitivity data is shown in Fig. 24. The dynamic stress-strain points at various strain rates and at various stress levels given by Fig. 23, may be plotted as shown. Thus a different dynamic stress-strain curve can be associated with each strain rate as indicated. Also included for comparison purposes is the tangent modulus line at zero stress to indicate the border line between the elastic and plastic strain region. Here again, as in the case of damping, it may be observed that at and below the cyclic stress sensitivity limit (29,000 psi), the strain is independent of strain rate and that above this limit both the plastic strain and the spread between the low strain rate and high strain rate curves increase with increasing stress.

Several of the observations made above with regard to frequency sensitivity as a function of stress are summarized in Fig. 25. This figure shows that (*a*) plastic strain, (*b*) change in damping with frequency, (*c*) change in dynamic modulus of elasticity with frequency, and (*d*) slope of semilog plot of total strain *versus* strain rate are all negligibly small below the cyclic stress sensitivity limit but increase with stress above this limit.

It might appear from Fig. 25 that the cyclic stress sensitivity limit defines the frequency sensitivity limit. However, it should be recalled that all data presented in Figs. 20 to 25 were procured on *different virgin* specimens exposed at *different* reversed *constant* stresses. Each specimen was brought to the stabilized damping region at constant stress amplitude and all damping and modulus measurements were taken at the same stress level as used in the stress history. Therefore, the

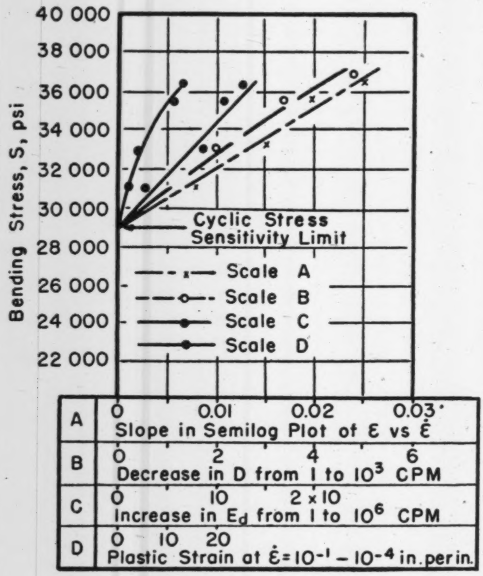

FIG. 25.—Frequency Sensitivity of Mild Steel Brought to Stabilized Damping Region at Stress of Measurement.

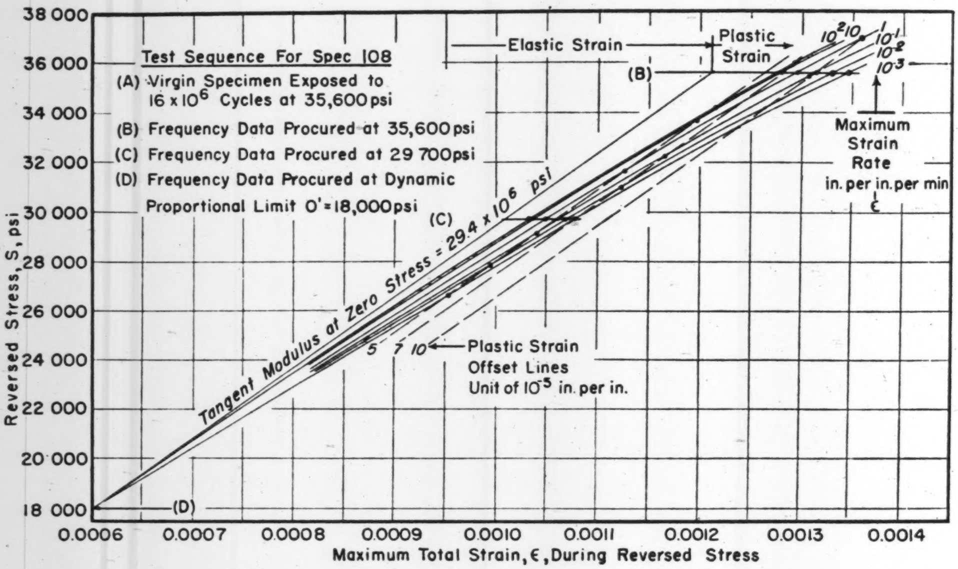

FIG. 26.—Effect of Strain Rate on Dynamic Stress-Strain Curve for Mild Steel Brought to Stabilized Damping Region at ±35,600 psi.

cyclic stress sensitivity limit is also the stress below which plastic deformation, *P*, is insignificant and above which *P* becomes large in the stabilized damping region. As shown in Fig. 17 only when the cyclic stress sensitivity limit has been exceeded during a stress history does significant plastic strain occur below this limit. It is consequently difficult to determine from the data presented thus far whether frequency sensitivity is primarily dependent on (*a*) how far the stress is above the cyclic stress sensitivity limit, or (*b*) the magnitude of the plastic strain component, *P*.

In order to clarify the above question specimen No. 108, previously discussed in connection with Fig. 17, was frequency tested as follows. After being brought to the stabilized damping region by 16 × 10^6 cycles at ±35,600 psi, frequency data were then procured at ±35,600 psi. These data were reduced to furnish the dynamic stress-strain points at ±35,600 psi as shown in Fig. 26. Similar frequency sensitivity data were then procured on this same specimen at ±29,700 psi as shown. During the 90-min period required to procure the ±29,700 psi data, no significant stress history effects were observed and the frequency sensitivity data were reproducible. Furthermore, reproducibility tests then made at ±35,600 psi indicated no stress history effects for this stress level and testing time. Tests were then made at and below the dynamic proportional limit Q' of ±18,000 psi and no measurable frequency sensitivity could be observed, as shown. Thus, the family of dynamic stress-strain curves diagrammed in Fig. 26 indicate the effect of strain rate on a specimen brought to the stabilized damping region at ±35,600 psi.

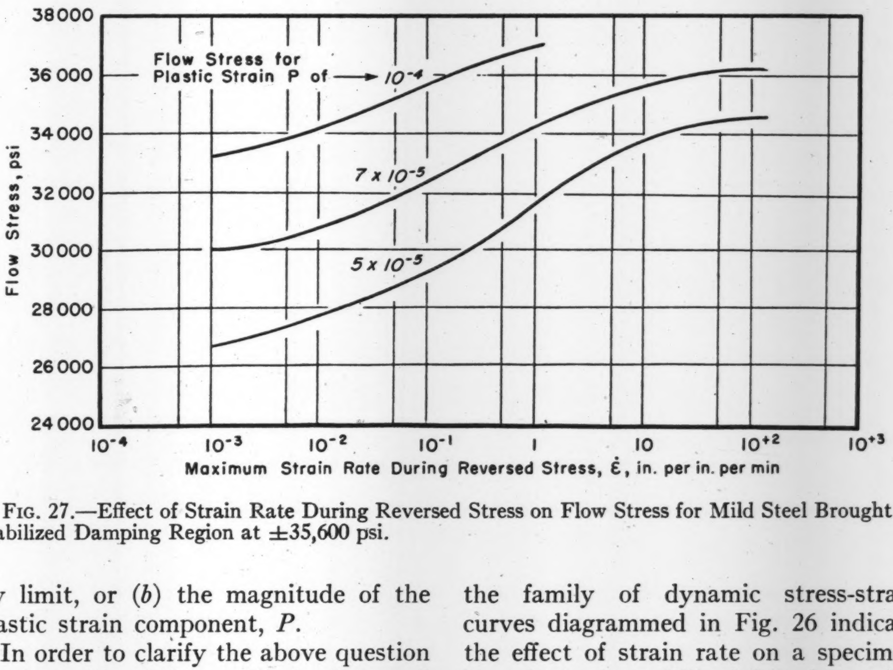

FIG. 27.—Effect of Strain Rate During Reversed Stress on Flow Stress for Mild Steel Brought to Stabilized Damping Region at ±35,600 psi.

In discussing the significance of these data, it should be mentioned that the dynamic proportional limit of this specimen after the ±35,600 psi stress history is approximately 18,000 psi as shown in Fig. 17. Therefore, in this case the dynamic proportional limit is below the cyclic stress sensitivity limit for virgin specimen (29,000 psi) and significant plastic strain P is displayed at and below this sensitivity limit. It may therefore be concluded from this test that the magnitude of plastic strain is probably more significant than how far the stress is above the cyclic stress sensitivity limit.

Summarizing, the data presented indicate that at engineering stress levels the frequency or strain rate sensitivity of damping and total strain depend primarily on the magnitude of the plastic strain component which persists during the reversed stress. In cases where the deformation is essentially elastic, the frequency effect is negligible. A quantitative analysis of these relationships is not planned until considerable more data are available.

The frequency sensitivity data presented herein may also be used to show the dependence of flow stress on strain rate as shown by the following example. If a series of offset lines for plastic strain $P = 5$, 7, and 10×10^{-5} are drawn parallel to the tangent modulus line at zero stress as shown in Fig. 26, the intersection of these offset lines and the dynamic stress-strain curves at the various strain rates provide flow stress data. Flow stress as used in this sense, defines the reversed stress amplitude at the various maximum strain rates indicated which will produce the specified reversed plastic strain. The strain rate dependency of flow stress is shown in Fig. 27 for three magnitudes of plastic strain.

The association of flow stress relationships so procured with those procured under unidirectional tension at various strain rates (such as occur in creep, stress rupture, static tension, and impact tests) constitutes a problem for future study. The wide range of strain offered by the comparatively simple and fast rotating-beam test may be an important advantage in justification of further emphasis on procuring the type of data presented in this paper.

SUMMARY AND CONCLUSIONS

The damping and dynamic modulus properties of mild steel procured during reversed bending fatigue tests up to 120 per cent of the fatigue limit are presented and analyzed. The concept of the cyclic stress sensitivity limit below which damping is insensitive to stress history, rest, and frequency is further discussed. Data on the effect of stress history, rest, and frequency in cyclic stress tests above this limit are also presented.

Observations concerning stabilized damping points beyond which damping is practically insensitive to additional stress history are discussed and included in an extended S-N fatigue diagram. This S-N-N diagram is subdivided into four regions in order to classify the various types of damping characteristics. Also demonstrated in the S-N-N diagram is the possibility that the stabilized damping line may establish the knee of the S-N curve.

A new type of graphical representation (called the S-N-D diagram) is introduced to summarize fatigue and damping data and also to indicate, through isodamping contour lines, the variation in damping as a function of stress magnitude and stress history. Similar data are also presented for the dynamic modulus of elasticity.

Preliminary tests performed on the effects of rest from cyclic stress after attainment of stabilized damping indicate that damping does not change significantly until after approximately 200 min of rest. Prolonged rest, however, causes a large decrease in damping, as much as 50 per cent after ten days.

The dynamic stress-strain relationships of mild steel were determined under reversed stress of gradually increasing magnitude in order to establish proportional limits, plastic strain components, etc. Observations made on these dynamic stress-strain curves lead to the following conclusions:

A. Below the cyclic stress sensitivity limit:

1. Both the static and dynamic stress-strain curves coincide.
2. Both are independent of stress history.
3. Neither the static nor the dynamic proportional limit is reached.

B. Between the cyclic stress sensitivity limit and the fatigue limit:

1. Both the static and dynamic stress-strain curves coincide *for the virgin specimen.*
2. Neither the static nor dynamic proportional limit is reached *for the virgin specimen.*
3. Under sustained cyclic stress:
    (*a*) Both the static and dynamic proportional limit decrease, the former more than the latter.
    (*b*) The static and dynamic stress-strain curves gradually deviate from each other.
    (*c*) The plastic strain component increases up to its limiting value at the stabilized damping point. The static plastic strain increases more than the dynamic value.
    (*d*) In spite of the above changes, the initial tangent modulus of elasticity remains unchanged.

C. Above the fatigue limit, the behavior is similar to, but more pronounced than, that discussed under "B" above. In addition

1. The dynamic proportional limit *of the virgin specimen* is near the fatigue limit.
2. The tangent modulus at zero stress does not remain constant but gradually decreases under sustained cyclic stress.

The association of the dynamic proportional limit with the fatigue limit may be significant, at least for limited groups of materials, from point of view of developing an accelerated fatigue test.

A series of tests on the effects of frequency of cyclic stress on damping and dynamic modulus indicate the following conclusions for the range from 0.05 to 10,000 cpm:

A. *Damping and dynamic modulus of mild steel are insensitive to frequency below the dynamic proportional limit.*—In view of the variation in dynamic proportional limit with stress history, as discussed previously, it may be concluded that:

1. For a *virgin* specimen frequency insensitivity persists up to the stress at the fatigue limit.
2. Specimens exposed to stress history display frequency insensitivity only if no part of the stress history exceeds the cyclic stress sensitivity limit.
3. Specimens exposed to a stress history above the cyclic stress sensitivity limit may become frequency sensitive at rather low stresses, well below the cyclic stress sensitivity limit.

B. *Damping and dynamic modulus are dependent on frequency at stresses above the dynamic proportional limit.*—The magnitude of this frequency effect increases with increasing plastic strain component. Damping increases slightly up to approximately 0.5 cpm and decreases as much as 50 per cent with increasing frequency beyond this point. Dynamic modulus values display a continuous increase with increasing frequency.

The frequency sensitivity of the dynamic modulus of elasticity, as discussed above, may be interpreted in terms of strain rate *versus* total strain relationship. These data may also be converted to a flow stress basis.

Acknowledgments:

This project was sponsored by the Office of Naval Research, whose fine cooperation is greatly appreciated.

Several of the authors' associates were

very helpful in this work. L. J. Demer aided in the analysis of the data and its method of presentation and S. Tomkinson, M. Linza, and P. Madonna did most of the experimental work and reduced a large portion of the data.

REFERENCES

(1) B. J. Lazan, "A Study With New Equipment of the Effects of Fatigue Stress on Damping Capacity and Elasticity of Mild Steel," *Transactions,* Am. Soc. Metals, Vol. 12, p. 499 (1950).

(2) B. J. Lazan, "Dynamic Creep and Rupture Properties of Temperature Resistant Materials Under Tensile Fatigue Stress," *Proceedings,* Am. Soc. Testing Mats., Vol. 49, p. 757 (1949).

(3) L. Bairstow, "The Elastic Limits of Iron and Steel Under Cyclical Variation in Stress," *Philosophical Transactions,* Royal Soc., Vol. A, p. 210 (1910).

(4) H. J. Gough, "The Fatigue of Metals," Van Nostrand Co., New York, N. Y. (1926).

(5) E. Orowan, "Theory of Fatigue of Metals," *Proceedings,* Royal Soc., London, Vol. 171A, p. 79 (1939).

(6) J. Bauschinger, "Change of Position of the Elastic Limit Under Cyclical Variations in Stress," *Dingler's Polytechnisches Journal,* Bd. 224; Cwilingenieur, 1881; metthlg. des mechanisch-Technischen Laboratoriums in Munchen, Heft XIII, XXV.

(7) F. E. Rowett, "Elastic Hysteresis in Steel," *Proceedings,* Royal Soc., London, Vol. 89, p. 528 (1913).

(8) A. L. Kimball and D. E. Lovell, "Internal Friction in Solids," *The Physical Review,* second series, Vol. 30, pp. 948–959 (1927).

(9) C. Zener, "Elasticity and Inelasticity of Metals," University of Chicago Press (1948).

(10) J. M. Robertson and A. J. Yorgiadis, "Internal Friction in Metals," *Journal of Applied Mechanics,* September, 1946, p. A-1738, A-182.

(11) J. D. Eshelby, "Dislocations as a Cause of Mechanical Damping in Metals," *Proceedings,* Royal Soc., London, Vol. 197A, p. 396 (1949).

(12) J. S. Koehler, *Technical Report No.* 4, Contract N 6 ori-47, Task Order 1, NR015-001, November, 1949.

(13) A. Nadai, "Stephen Timoshenko 60th Anniversary Volume," MacMillan Company, Inc., New York, N. Y., p. 155 (1938).

DISCUSSION

MR. ALEX YORGIADIS[1] (*by letter*).—This paper shows, if such evidence were still needed, that materials in general, and mild steel in particular, are severely affected by cyclic stresses of certain intensity, and that the general behavior of materials under these conditions is so involved that a multitude of variables must be considered in order to obtain reliable data.

In presenting their results, the authors have had to devise new terms, either because existing ones were not satisfactory, or because none existed. For example the authors use the term "stress history" to define cycles of stress applied to a specimen prior to a particular measurement. One of the authors in a previous publication,[2] used the term "cold working" to represent the same thing. In some respects, "cold working" is descriptive in that it represents the straining of a material to a degree that changes its physical condition and properties. Nevertheless, this is usually associated with manufacturing processes of forming, and the stresses involved are high, usually beyond the yield point, and are applied very few times, usually once or twice. The writer therefore agrees that the present expression used by the author is an improvement over the one he used in the past. The authors have introduced a number of other new terms associated with this field of testing. It is suggested that these be studied so as to standardize the nomenclature.

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[1] Chief Engineer, Sonntag Scientific Corp., Greenwich, Conn.

[2] B. J. Lazan, "Some Mechanical Properties of Plastics and Metals Under Sustained Vibrations," *Transactions,* Am. Soc. Mechanical Engrs., Vol. 65, pp. 87–104 (1943).

The "stabilized damping behavior" of mild steel is in some respects similar to the behavior of magnesium alloys. Several years ago, Kutsay and the writer conducted torsion tests and reported on the damping capacity of two commercial grades of magnesium alloy rods as affected by "stress history."[3] While the material and mode of stressing were different, the results sought were the same. The curves reported in Figs. 4 and 5 of this reference are similar to those in Fig. 7 of the paper under discussion. In all of these, the X-axis represents stress on a uniform scale, the Y-axis represents damping energy on a logarithmic scale, and the curves show the damping of a given specimen with a prescribed stress history. In addition, Fig. 10 of this reference is similar to Fig. 6 of the paper. The damping in the magnesium alloys tested decreased due to cyclic stress, which is opposite to what the authors reported for mild steel. The mild steel used by the authors was annealed, while the magnesium specimens were extruded rods, with internal strains. However, just as in the case of the authors' tests, a certain "equilibrium" was reached after a given number of stress cycles, beyond which the damping did not change by further stress cycles.

The authors report that cyclic bending stresses below 29,000 psi have no effect on damping and dynamic modulus of elasticity of the material tested, namely, SAE 1020 steel. Data previously reported by Lazan[2] do not show the existence of such a limit for SAE 1015 stressed in torsion. Figure 5 of this earlier paper shows an entirely different behavior in that at low stresses damping decreases as a result of cyclic stressing. At the high stress levels damping increased as reported now. The transition stress was in the vicinity of 22,000 psi shear stress. The writer has reported some data of damping of SAE 1025 steel under tension-compression loading.[4] At 29,000 psi alternating stress, the observed damping energy was approximately 1.6 in.-lb per cu in. per cycle. The authors obtained a value of 0.4 in-lb for their material at this same stress. Admittedly the tests were performed on slightly different materials, and the test errors were not entirely eliminated, particularly in the tests by the writer which were not as elaborately prepared as those by the authors. Some possible causes of differences that cannot be accounted are as follows:

In the rotating-beam tests by the authors, the various circumferential fibers are subjected to different instantaneous stresses even though eventually each fiber is subjected to the full cycle of stress. The fact that adjacent fibers are at different stresses may produce a different over all behavior than that produced in a tension-compression test where all the fibers in the specimen are at the same instantaneous stress at all times.

Tubular bending specimens cannot be uniformly stressed since the stress varies in proportion with the radial location of a given fiber. By using thin-walled tubes, this effect can be minimized, and, as noted by the authors, the stress in their test specimens varied between the reported value at the outer surface, to 90 per cent of this stress at the inner surface. Referring to Fig. 7 of the paper, a stress of 29,500 psi actually represents a stress distribution varying from 29,500 to 26,500 psi. Only one-sixth of the volume of the specimen is stressed above the sensitivity limit of 29,000 psi, so that only one-sixth of the specimen is subject to a change in damping at this point. The total damping energy is distributed over the entire volume of the specimen, so that the quantities reported are not representative of the true quantitative basic behavior of an element of the material, but only represent some average values. Since the average damping increases 3-fold at this stress, the localized damping of the layers stressed between 29,000 and 29,500 psi must, on an average, increase 18 times. At 32,200 psi stress, the entire volume of the specimen is above the sensitivity limit of 29,000 psi, so that all the fibers are subject to a change in damping. The chart shows an increase in damping by a factor of only 8 or 9. The writer would like to have the author's explanation of this apparent discrepancy.

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[3] A. U. Kutsay and A. J. Yorgiadis, "On the Torsional Damping Capacity of Solid Magnesium Alloy Rods as Affected by Cold-Working," *Journal of the Aeronautical Sciences*, Vol. 10, pp. 303–310 (1943).

[4] J. M. Robertson and A. J. Yorgiadis, "Internal Friction in Engineering Materials," *Journal of Applied Mechanics*, September, 1946, p. A-173 to A-182.

At least one very immediate practical value of the authors' data relates to fatigue testing. A large percentage of fatigue tests are conducted on repeated deflection or repeated strain type of fatigue machines, but the results are invariably reported in terms of repeated stress, measured at the start of the test, or some selected points in the test. The ASTM Manual on Fatigue Testing considers such procedures as acceptable. The authors' data in Figs. 11 and 12 show most conclusively that it is not justified to assume a constant modulus of elasticity for mild steel subjected to stresses high enough to produce fatigue failures. At the fatigue limit, the modulus is seen to drop to 90 per cent of its initial value, while at 40,500 psi stress corresponding to a life of 100,000 cycles, the modulus drops to less than 75 per cent of its initial value. Such reductions in modulus would cause severe reductions in stress for a given strain, rendering fatigue data on constant strain machines very inaccurate.

MESSRS. B. J. LAZAN AND T. WU (*author's closure*).—Mr. Yorgiadis has inquired about the discrepancies in damping data on steel tubing in torsion reported by one of the authors in 1943[2] and the data presented in this paper on a bending specimen machined from a similar material, hot-rolled mild steel. The discrepancies in the earlier work were of two types: (1) the damping was significantly higher, and (2) the damping under sustained cyclic stress decreased instead of increasing. It is probably true, as Mr. Yorgiadis points out, that the differences in material and type of stress (torsion *versus* bending) cannot explain the differences. Mr. Yorgiadis offers two possible reasons for this difference: (1) stress phasing effects during rotating bending, and (2) the stress variation across the tubular wall. In the opinion of the authors, neither of these factors is as important in explaining this difference as the "stress-history" or "cold work" effect discussed below.

During the earlier work on steel tubing in torsion the study of the effects of stress history were in a purely exploratory stage; little was known concerning the existence of a stress-history effect, let alone its nature and magnitude. Furthermore, the equipment used in this earlier work did not enable suitable control on stress-history. As a result, the tubular specimen in torsion received a variable high-stress history prior to the test run plotted in Fig. 5 of this earlier paper. The effects of this prior high-stress history can easily explain the discrepancies cited above. Referring, for example, to Figs. 10 and 11 of reference (1) in the paper it may be seen that prior stress history at high stress levels may (1) significantly increase the damping (as shown also in this paper), and (2) may cause subsequent cyclic stress at a lower stress level to reduce the damping. Also the rest effects discussed in this paper (Fig. 15), which were not controlled in the earlier work, may exert additional effects which can partially explain the trends observed in the earlier work. The authors therefore believe that the data presented in this paper is much more reliable than that presented in earlier papers by one of the authors and others, because in the earlier work the full significance of stress history was not understood.

Mr. Yorgiadis also inquired about the apparent discrepancy in the data indicating the change in damping at a peak specimen stress of 29,500 psi as compared to 32,200 psi. His calculations are based on an extrapolation of the stress-history effect attributed to the difference in the peak stress of 29,500 psi and the cyclic stress sensitivity limit of 29,000 psi. As pointed out by the authors on this paper, damping is an extremely sensitive property which depends on specimen finishing technique, etc., to a much greater extent than most other properties. Although the authors did not procure statistical data on the variation in the cyclic stress sensitivity limit among what should be identical specimens, the few observations made certainly indicate that a variation greater than the 2 per cent difference used by Mr. Yorgiadis in his extrapolation was to be expected. Thus, the authors do not consider the difference discussed by Mr. Yorgiadis to be a discrepancy but rather an already acknowledged indication of the variability of damping data. Studies of published literature indicate that although static strength properties show good consistency (and agreement within 2 per cent even in static work is considered good in many cases), fatigue data and especially damping data show considerably larger scatter.

I am glad that Mr. Yorgiadis has emphasized the importance of considering the variability in dynamic modulus of elasticity in the interpretation of constant-deflection type fatigue data. This important factor is still frequently overlooked in fatigue research.

FATIGUE STRENGTH OF BALL BEARING RACES AND HEAT-TREATED 52100 STEEL SPECIMENS*

By Haakon Styri[1]

Synopsis

Numerous fatigue tests on ball bearings have shown that the lives of individual bearings before failure by spalling or flaking, show a scatter over a wide range of 1 to 50 or more. It has also been established that such failures usually start from points below the contacting surfaces and that considerable variation of heat treatment has little influence on this scatter. The median life of ball bearings varies inversely about as the cube of the bearing load P, and since the mean normal pressures at the contact points are proportional to $\sqrt[3]{P}$ the lives of the bearings therefore vary inversely as about the ninth to tenth power of these pressures.

In order to study the relation of load to life, as well as the effect of heat treatment on simple specimens, tests were run on ring-type, rotating-beam-type, Krouse-type, and torsion-type specimens. With these types of specimens, the surfaces could be given as fine a finish as desired and would therefore be practically eliminated as a cause of failure and since the specimens were made from steels of highest commercial quality, major defects in the steel could also be considered eliminated. However, it is known that even quite small inclusions may be starting points for cracks, and a vacuum-melted steel was obtained in order to study the effect of reducing slag inclusions to a minimum, but even with this steel the rotating-beam specimen gave wide scatter in life. Factors other than foreign inclusions must therefore provide starting points for failure and are most likely to be found in the anisotropic nature of the material and in strains and stresses caused by the transformation of austenite to martensite.

The relation of life to load in the different specimens varies inversely as about the twelfth power in the ring-type specimen, as about the fifteenth in the rotating-beam type, and as about the tenth power in the torsion test.

It is concluded that the fatigue failures in bearings start at local weakness points in a region under the rolling contact path where shear stresses are high.

Examples of the beginnings of structural disturbance and cracks are shown in photomicrographs at 100 and 1000 magnification.

It is well known that when a number of like articles such as electric lamp bulbs, or gears, or springs, or cutting tools, are subjected to service tests under exactly the same conditions, the lives before failure will vary greatly.

Fatigue Strength of Ball Bearings:

It is also well known that ball bearings are found to vary greatly in life before failure when run under identical conditions either in the field or in the laboratory. In the early 1920's several articles describing fatigue tests on ball bearings were published (**1, 2, 3**)[2], and since then more evidence has been published in various books (**4, 5, 6**). The results of an extended series of tests on a deep groove type bearing, run in our laboratory during the last couple of years, are shown in Fig. 1. The No. 6207 bearing has a bore of 35 mm, outside diameter

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* Presented at the Fifty-fourth Annual Meeting of the Society, June 18–22, 1951.

[1] Consultant, Research and Development, SKF Industries, Inc., Philadelphia, Pa.

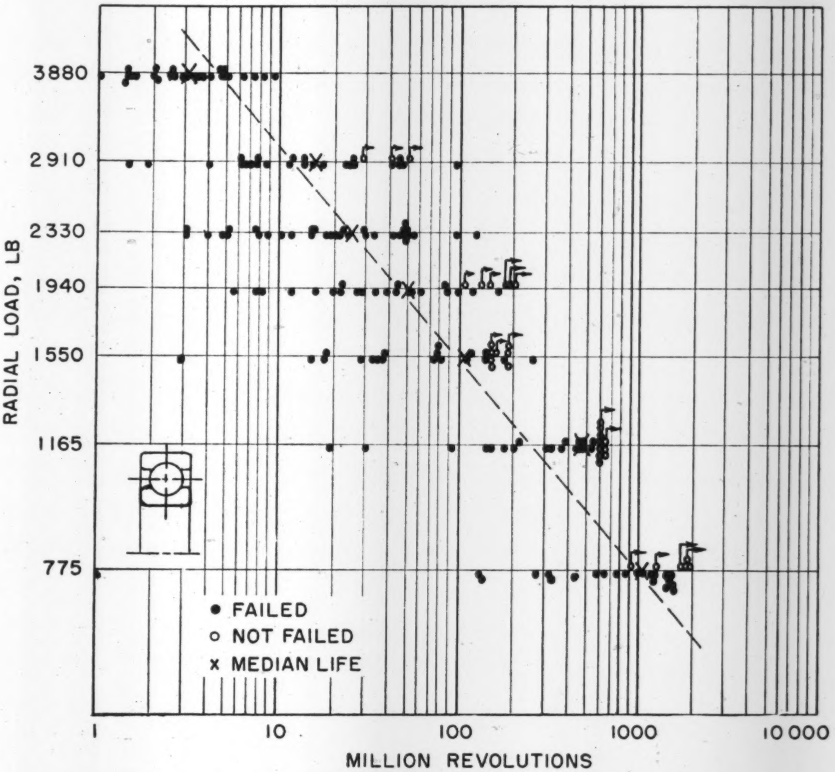

FIG. 1.—Endurance of Inner Races of No. 6207 Bearings.

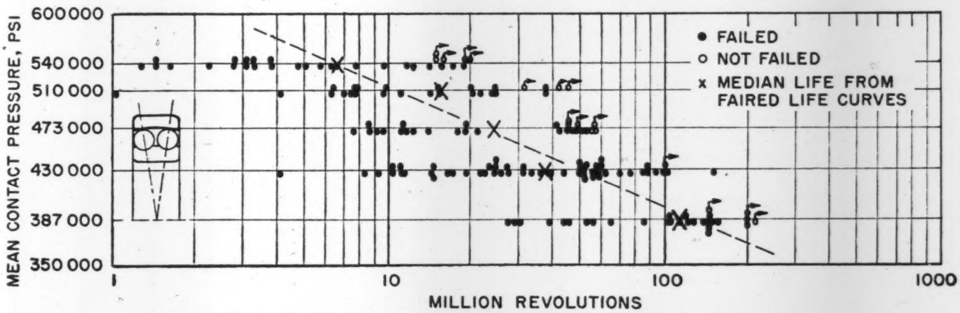

FIG. 2.—Endurance of Outer Races of No. 1207 Bearings.

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[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 696.

of 72 mm, and width of 17 mm. The load P used in each of the various test groups is given on the ordinate. The individual lives, L, obtained, are plotted as points along the abscissa. The log scales of the chart are designed to permit ready comparison of the slopes of the fatigue-life curves. The faired median life curve in this graph can be represented by a straight line of form

$$\frac{L_1}{L_2} = \left(\frac{P_2}{P_1}\right)^x$$

where the subscript 1 refers to life and load at one point and 2 at another and where x in this series of tests was found to equal 3.3. The scatter in life at the various loads is about the same, and there is no indication of any fatigue limit within the range of the tests. The failures plotted are all inner race failures.

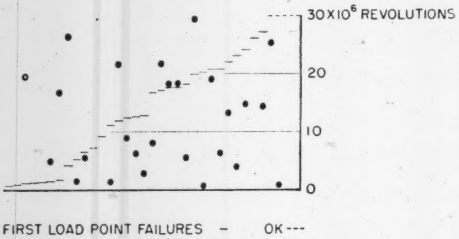


FIG. 3.—Endurance of Outer Races of No. 1309 Bearings Under Radial Load of 4240 lb.

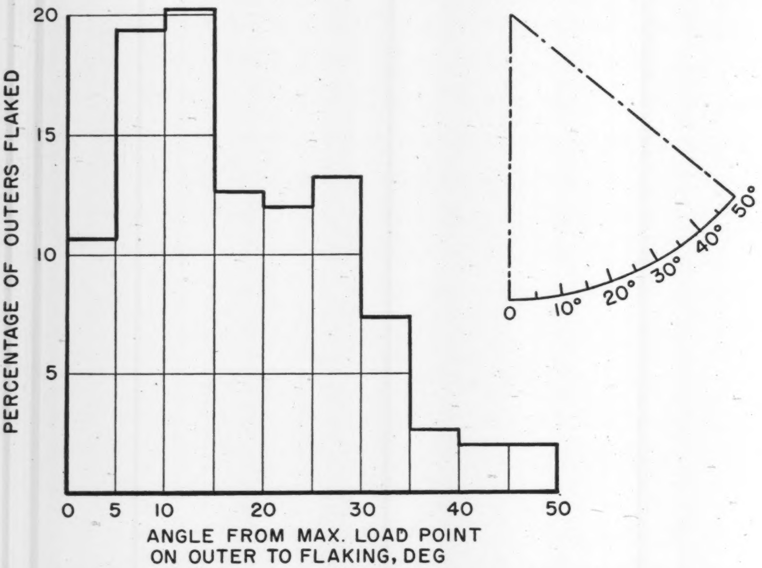
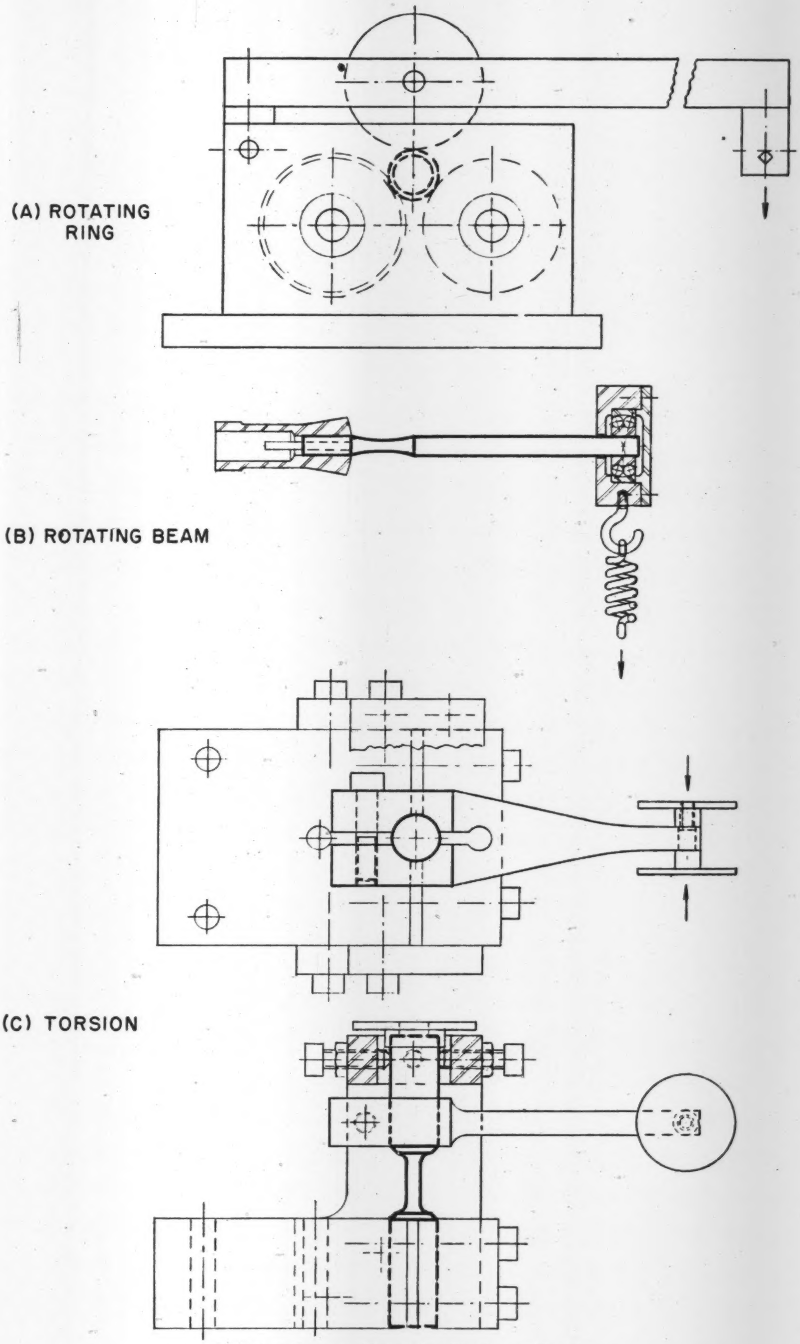


FIG. 4.—Location of Flakings in Outers of No. 1309 Bearings.

Another series of tests has been run on a self-aligning ball bearing, No. 1207 (Fig. 2), where the outer races were made of the same steels as those used in No. 6207 bearings. The loads selected are indicated on the ordinate as the mean normal contact pressure, σ, for the most heavily loaded ball against the outer race which, according to Hertz, is proportional to $\sqrt[3]{\frac{P}{d^2}}$, or P is proportional to $\sigma^3 d^2$ where P is the load, d the ball diameter. The individual lives are plotted on the abscissa, and the median fatigue failures of each group are indicated. A faired straight line for this median life can be expressed as:

$$\frac{L_1}{L_2} = \left(\frac{\sigma_2}{\sigma_1}\right)^9$$



(A) ROTATING RING

(B) ROTATING BEAM

(C) TORSION

FIG. 5.—Specimens Used in Fatigue Testing.

If, instead of the mean contact pressure σ, the load P on the bearing had been indicated on the ordinate, the life-load relation would be expressed by

$$\frac{L_1}{L_2} = \left(\frac{P_2}{P_1}\right)^3$$

These two tests show scatter of lives at each load about the same in a ratio of 1 to 50 or more.

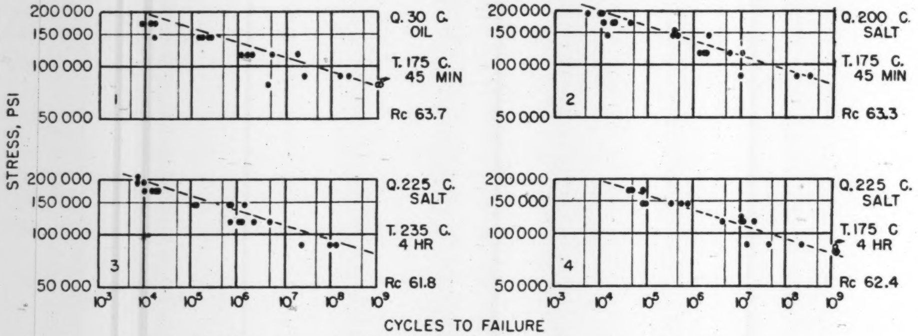


FIG. 6.—Endurance of SAE 52100 Steel Rotating-Ring Type Specimens.

An interesting experiment has been performed a number of times with self-aligning ball bearings. A group of 25 to 30 bearings were tested and a normal life curve for that test condition established. Thereafter the same outer races were refitted with new inner races and balls, and retested with the load directed against the opposite side of the outer race.

The lives of the outer races under this second test condition showed no individual relation to those of the previous test but failed with completely random distribution of lives, compared with the original lives, although the average was within the probable average of the group (Fig. 3). Another point of interest is that the location of the starting point of failure is not always at the most heavily loaded point, but is very often away from this point in the direction of the ball path (Fig. 4).

During many years of testing bearings, various means have been tried to reduce the scatter. The greatest efforts have been spent in studying the effect of various heat treatments and related microstructure, but while much has been learned about their influence on the level of fatigue life, no significant reduction in scatter has been obtained. It has not been possible to get noticeable reduction in scatter by using the cleanest steel obtainable. In all tests the failure starts from one point or at most from two or a very few points, even in bearings under pure thrust where all balls are subject to the same load.

Obviously there is a weakest spot in the track that provides the starting point for the failure.

Since we could not make any satisfactory headway by analyzing such a complicated mechanism as a bearing, attempts were made to develop some simple specimen for fatigue testing, on which we could study the effect of variation in a single factor of influence such as steel analysis, heat treatment, or surface finish. For this purpose four different type specimens and testing methods were used.

FATIGUE STRENGTH OF SIMPLE SPECIMENS

Ring-Type Specimens:

The first of these was a ring-type specimen which has been previously described (**7**), the details of which were worked out by the late W. F. Titus. The ring was loaded from the outside at three points 120 deg apart with the result that maximum tension stresses developed on the inside surface of the ring at these contact points at a rate of 14,000 cpm. (Fig. 5(*a*)). The original thought in developing this specimen was to find a suitable means of comparing steel quality, and a great number of tests have been run with different steels and different heat treatments. Results from the most recent series of tests are shown in Fig. 6.

TABLE I.—HEAT TREATMENTS.

Specimen type	Figure	Graph	Hardening Temperature, deg Cent	Quenching			Tempering		Rockwell Hardness, C Scale
				Medium	Temperature, deg Cent	Time	Temperature, deg Cent	Time	
Rotating ring	6	1	850	oil	30	15 sec	175	45 min	63.7
		2		salt	200	3 min	175	45 min	63.3
		3		salt	225	45 min	235	4 hr	61.8
		4		salt	225	45 min	175	4 hr	62.4
Rotating beam, SAE 52100 steel	7	1	850	oil	30	30 sec	175	45 min	63.5
		2		salt	170	3 min	175	45 min	63.9
		3		salt	220	90 min	175	45 min	61.8
		4		salt	220	90 min	240	4 hr	61.8
		5		salt	270	1 hr	175	45 min	58.5
		6		oil	30	30 sec	300	45 min	59.4
		7		normalized					38.1
		8		annealed					17.0
Rotating beam vacuum melted, SAE 52100	8	1	850	oil	30	30 sec	175	45 min	63.3
		2		salt	170	3 min	175	45 min	63.7
		3		salt	270	60 min	175	45 min	57.3
		4		oil	30	30 sec	300	45 min	55.9
		5		salt	170	3 min	490	45 min	42.5
		6		salt	210	3 min	530	45 min	30.0
Torsion specimens	9	1	850	oil	30	1 min	175	45 min	62.2
		2		salt	185	3 min	175	45 min	62.4
		3		salt	230	3 min	175	45 min	62.4
		4		salt	230	50 min	175	45 min	60.2
		5		salt	230	50 min	235	4 hr	59.2
		6			miscellaneous		heat treat		45.0
		7		oil	emerged at 50 C		575	30 min	32.4
		8		oil	emerged at 150 C		700	30 min	27.9

The rings were machined from one tubing of SAE 52100 steel, heat treated as shown in Table I, ground to size, and finally the bores were honed to better than 3 microinch rms finish and the corners slightly rounded. The results of the test showed that none of the various heat treatments, resulting in hardnesses of about 60 Rockwell C or over, gives a greater level of fatigue life than the others and that the scatter of life at the different loads is of the same magnitude. This specimen, with the dimensions given, could not be used for tests at hardness levels below 50 Rockwell C because first failures frequently occurred on the outside instead of on the inside, particularly at the lighter loads, due to contact with the loading rollers. No indication of a fatigue limit is evident up to about 400 million cycles, although 80,000 psi seems probable for hardnesses over 60 Rockwell C. In practically every case there was only one starting point of failure. Unfortunately it was not possible to stop the test quickly enough after failure, with the result that the fractures were usually badly damaged. However, occasionally, clean fractures were obtained, and then a nucleus for the starting of the failure was evident.

Krouse-Type Specimens:

A Krouse-type specimen was used in some tests run by us eight years ago on special bearing steel samples and were described by the author in comments to the paper by Tarasov and Grover (8). Since then additional tests have been run, but unfortunately mixed samples of steel had been obtained from our source of supply so that results are not sufficiently reliable to determine the load life curve. The Krouse specimen compared with the ring type, had an advantage in that the fracture was almost always undamaged from rubbing and the starting point could therefore be observed easily. It was located entirely at random over the fractures, usually occurring at or very near the surface at the point of maximum stress, but sometimes originating from visible defects below the surface.

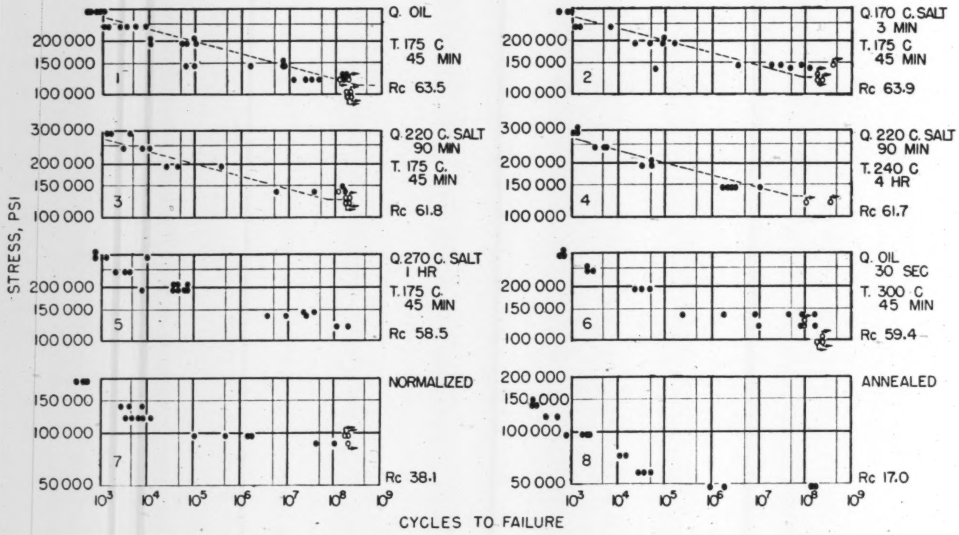


FIG. 7.—Endurance of SAE 52100 Steel Rotating-Beam Type Specimens.

Rotating-Beam Type Specimens:

Because the Krouse specimen was rather expensive to make and the tests slow, a third method was developed which permitted the use of a simple testing machine with relatively rapid testing and of a simple specimen of more uniform material. A rotating-beam type specimen, Fig. 5, with a necked-down section was selected with the purpose of limiting the start of failures to a small area of the specimen in order to simulate conditions in ball bearings where the failures start in the small areas subjected to load.

Several available grinding spindles with carefully centered chucks that were well finished in the bore were used to hold the specimens. A self-aligning ball bearing was mounted on the outer end of the specimen, and the outer race of the ball bearing was fitted into a housing which was damped sidewise in Keldur-lined guides and to which proper weights were hung through a soft spiral wire spring to give the desired bending stress. This damping was found necessary because the slender specimen otherwise had a tendency to whip when accelerated to the operating speeds of 13,000 rpm. When the specimen broke, a switch was actuated, cutting off the power to the driving motor. A revolution counter on the motor permitted calculation of the number of cycles run to failure.

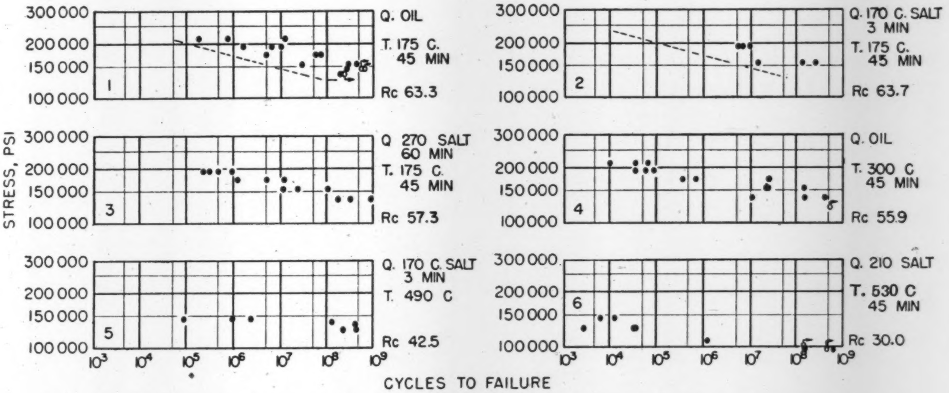


Fig. 8.—Endurance of Vacuum-Melted SAE 52100 Steel Rotating-Beam Type Specimens.

A great many tests were run with specimens from a number of different bearing steels and with a great variety of heat treatments. In one series (Fig. 7) the specimens were all taken from one coil of wire, cut to length, straightened, heat treated, and centerless ground. The neck was then rough and finish ground and given a surface finish of better than 3 microinches rms by careful lapping in the axial direction.

The lives of the specimens hardened in different ways to 60 Rockwell C or higher do not differ much in range or in slope against applied loads. The scatter in lives at the higher loads is somewhat less than at the lower loads, but more tests were run at the lower values. As a possible fatigue limit is approached, the scatter band widens as described earlier by J. O. Almen (9). Obviously, some specimens are stronger than others at the critical section and carry more load for the same fatigue life, or have longer life carrying the same load. Local weakness was also apparent in the testing of ball bearings and resulted in a wide scatter in life, depending on the proximity of the weak spot to the contact point.

It is generally considered that slag inclusions are major defects from which fatigue failures start. In an effort to reduce the effect of inclusions as much as possible and particularly to learn if the scatter would be improved a test was run on specimens made of vacuum-melted SAE 52100 steel, kindly furnished us by Babcock & Wilcox Co. Necked-down rotating-beam specimens were made from one coil of wire and given various heat treatments, with results shown in Fig. 8. A wide scatter appears here also, in spite of the great reduction in size of foreign inclusions. In both Figs. 7 and 8 reference lines are drawn, representing an approximate load-life curve for the median lives of standard SAE 52100 specimens with hardness $\geqq$ 60 Rockwell C, but such lines are not drawn in graphs of lower hardness.

It is of interest to note that the fatigue lives of the vacuum-melted steel are considerably greater than for the standard SAE 52100 steel for hardness ≧ 60 Rockwell C. It is not certain whether this will also be the case for a fatigue limit, since so far no well-defined fatigue limit has been established. Possibly the limit for rotating beam of vacuum-melted steel may be 130,000 psi, about 5000 psi higher than for the standard steel.

Both the standard and the vacuum-melted steel specimens with hardness between 57 to 40 Rockwell C have lower lives than the harder specimens, when tested at stresses higher than the fatigue limit.

For still lower hardnesses, definite fatigue limits were found. The standard steel, SAE 52100 normalized to 38 Rockwell C, gave about 90,000 psi and annealed to 17 Rockwell C, about 50,000 psi. The vacuum-melted steel which was quenched and tempered to 42 Rockwell C gave 125,000 psi and tempered to 30 Rockwell C, 100,000 psi.

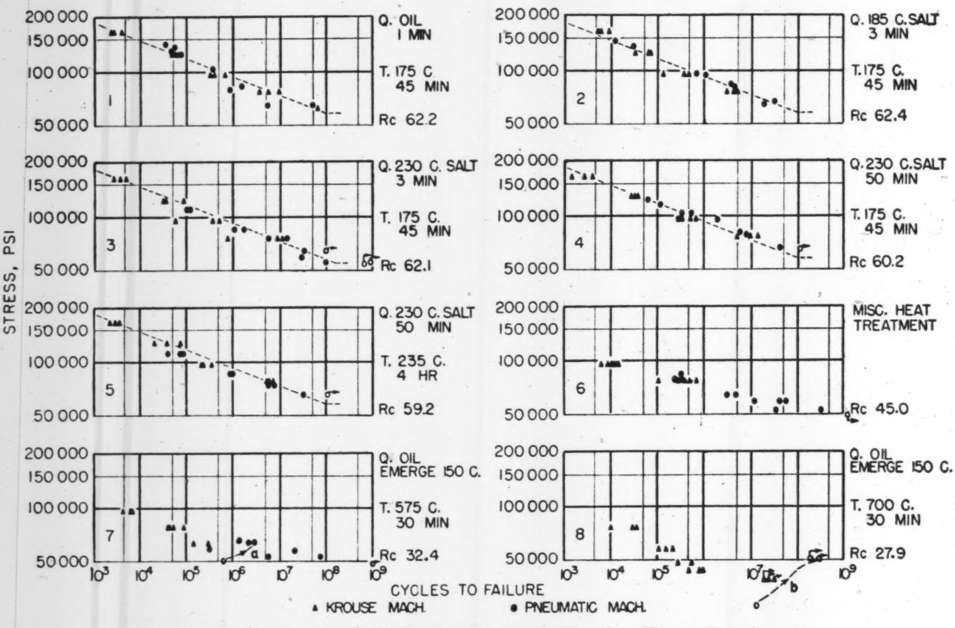


FIG. 9.—Endurance of SAE 52100 Steel Torsion-Type Specimens.

Each of the three specimen types tested for endurance at high hardness gave relatively high scatter in lives when tested under alternating tension and compression in bending.

Torsion-Type Specimens:

It was therefore of interest to see whether another type of specimen operating under alternating shear stresses would give different results. A torsion specimen shown in Fig. 5(*c*) was designed, made from wire and heat treated after necking-down by turning, and finished by careful grinding and polishing. Alternating torsion was run on some specimens in a Krouse machine which gave a constant amplitude of deflection and where the angle of twist was determined by direct load calibration. At failure, a mechanism separated the broken pieces and simultaneously cut the power supply to the driving motor. The lives of these specimens are plotted as solid triangle in Fig. 9. Lives of unbroken specimens are shown with open triangles.

Other specimens were tested at higher torsional frequency of about 14,000 per min in an air-driven machine built according to the principle used by F. B. Quinlan (10), but somewhat modified (Fig. 5(*c*)). The details of this machine were worked out by T. Morrison. The lives of the broken specimens are marked solid circle in Fig. 9, the lives of unbroken specimens with open circles.

The life curves for the torsion specimens do not differ much for different methods of quenching and tempering that result in hardness values of 60 Rockwell C or higher, and no fatigue limit is indicated for these specimens for lives up to 10^9 cycles, although one may be expected just below 60,000 psi.

When specimens had been tempered to 45 Rockwell C the lives to failure were shorter than for harder specimens, the slope of the life curve was less, and a fatigue limit of about 50,000 psi was indicated.

Specimens of 32 Rockwell C tested in the Krouse machine gave a fatigue limit of 50,000 psi, but specimens of this hardness could not be run satisfactorily in the pneumatic machine. When applying an excess air pressure, the angle of twist expected of a fully elastic specimen could not be obtained because of a partial plastic yielding which caused internal damping. The specimen would gradually work-harden and therefore sustain higher and higher angles of twist and loads before failure as indicated by *a* in graph 7 of Fig. 9. Specimen of 28 Rockwell C *b* in graph 8 of Fig. 9 showed this effect to a more pronounced degree.

There is relatively less scatter in life in the torsion fatigue test on specimens of high hardness, as compared with the rotating-beam fatigue test. Probably an applied shear stress nearer to the possible fatigue limit would show wider scatter as indicated by test on specimens of lower hardness.

All four tests show, however, that we should expect a scatterband with a lower and a higher limit for fatigue life, as previously indicated by Almen (9) and Dolan (15), and a lower and a higher fatigue limit, if and when such is found at light enough loads. The individual life would depend on presence or absence of defects or weak spots in small elements of the critical area.

DISCUSSION

If we consider the relation of life to load in the methods described and select the median lives at the different loads, we find that a straight line can be faired through these points reasonably well in the log-log graph. In other words, the following formula,

$$\frac{L_1}{L_2} = \left(\frac{S_2}{S_1}\right)^x,$$

is roughly representative for all. The value of x is different for the types of specimens used:

ring specimens x	approximately 12
rotating beam x	approximately 15
torsion x	approximately 10

In Fig. 1 the value of x was found to equal 3.3.

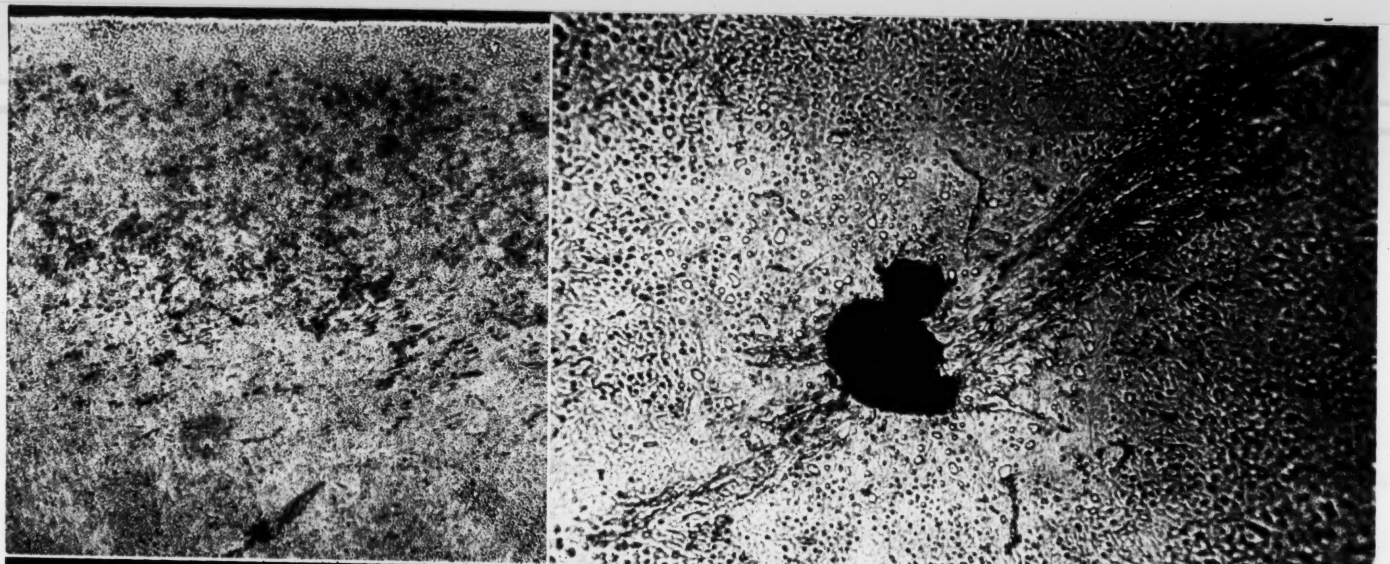
In Fig. 2 the value of x would be 3 if loads P had been used instead of contact pressures σ.

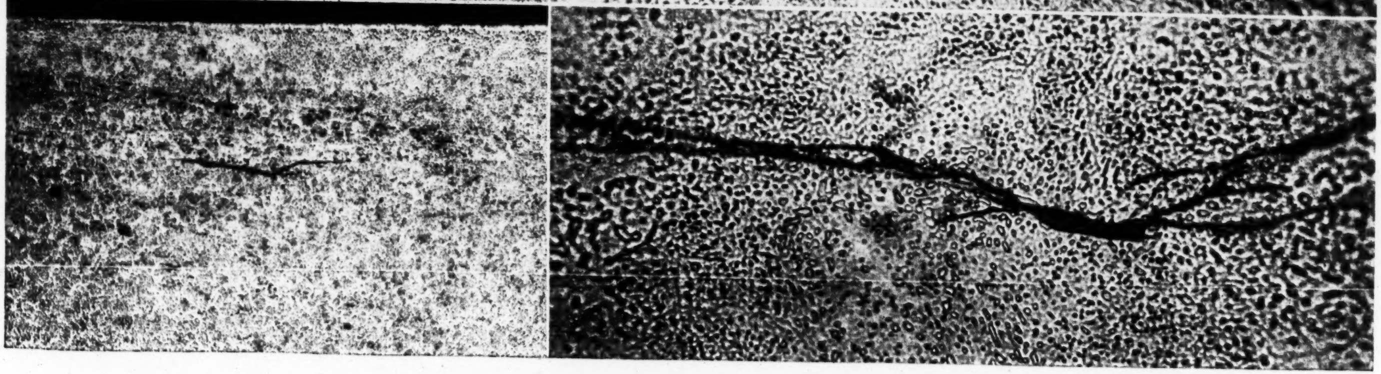
Numerous other fatigue tests run on ball bearings, here and abroad, generally agree that the 10 per cent, the median (50 per cent), and the average (about 60 per cent) lives before failure at different loads vary approximately inversely to the third power of the loads on the bearing (3, 6, 14).

In testing at the lighter loads, some wear in the bearings may influence the life adversely and selection of $x = 3$

× 100 × 1000

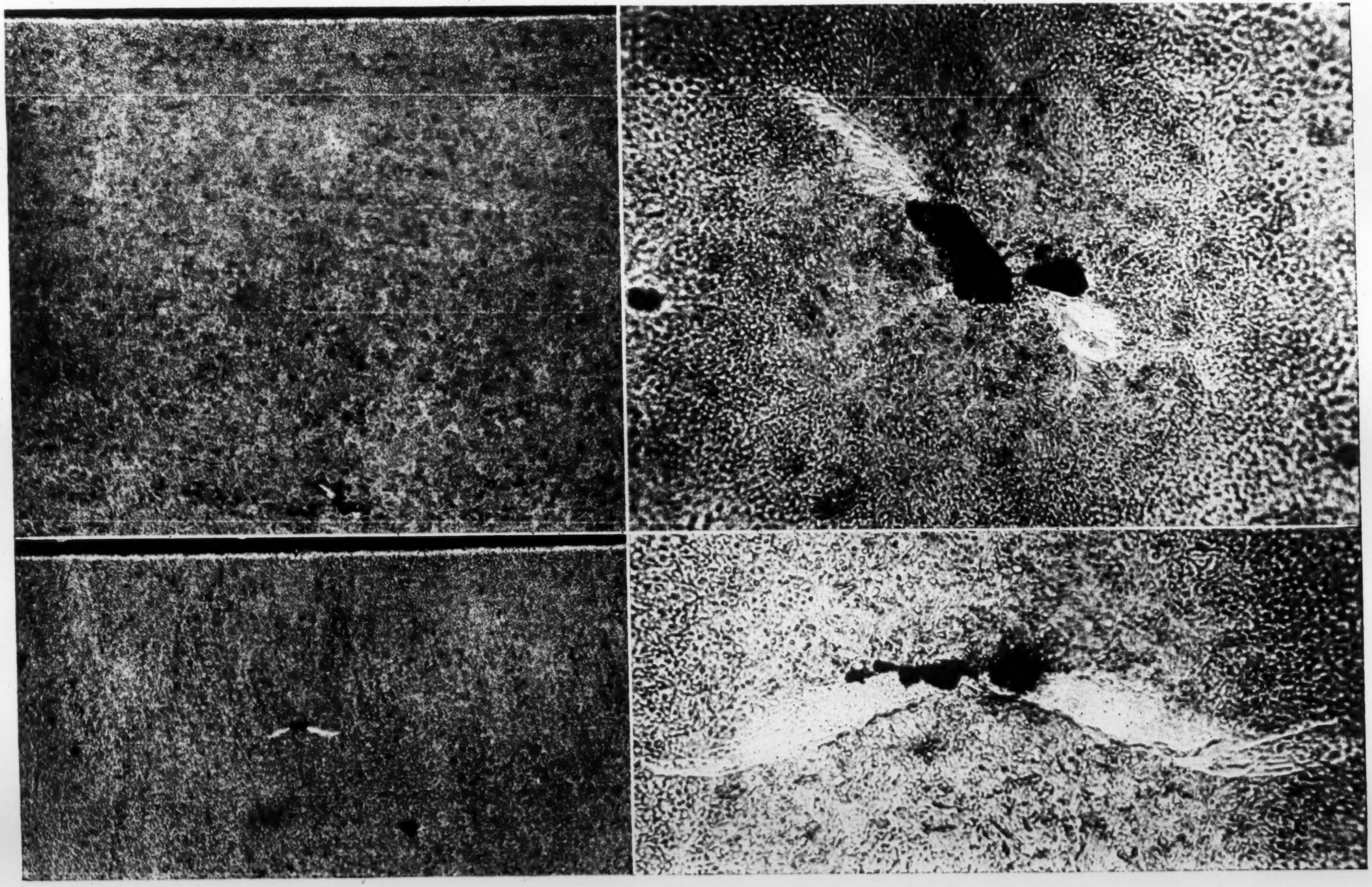
(*a*) Cuts taken at right angles to the axis.





(*b*) Cuts taken at right angles to the axis. Crack without slag inclusions.

(*c*) Cuts taken at right angles to the axis.



(*d*) Radial cut parallel to the axis

× 100 × 1000

FIG. 10.—Start of Failures.

instead of $x = 3.3$ is therefore preferred in catalogues of bearing capacities, in order to be on the conservative side.

By substituting mean normal pressures at contact points of the most heavily loaded ball for the bearing load, the lives vary approximately inversely as the ninth to tenth power of the loads.

This fact suggests that the cause of failure in ball bearings may be similar to that in torsion specimens, and that since the failures in the torsion tests in practically all cases started in the direction of alternating shear stresses, we may expect to find starting points for fatigue flakings in ball bearings in regions where relatively heavy shear stresses prevail (11). According to Lundberg and Palmgren (12) this region would be at a depth of one-third to one-half of the minor half-axis of the contact ellipse, and the magnitude of the shear stresses, which are supposed to cause the start of failure, are from 0.43 to 0.5 of the maximum pressure at the contact point. With a probable fatigue limit in torsion test $\overset{=}{<}$ 60,000 psi, a definite fatigue limit for ball bearings should not be expected at maximum contact pressures higher than about 120,000 psi. This would be of practical interest only in applications where bearings are made over-size for reasons other than to have them last longer than the equipment.

The author reported in 1925 and later (2, 13) that the flaking usually started below the surface where the ball track was located, and the cracks grew from there to the surface. A number of times since then this observation has been verified by taking sections of races crosswise or lengthwise of the ball tracks at points where no cracks were observable on the surface after a prolonged fatigue test.

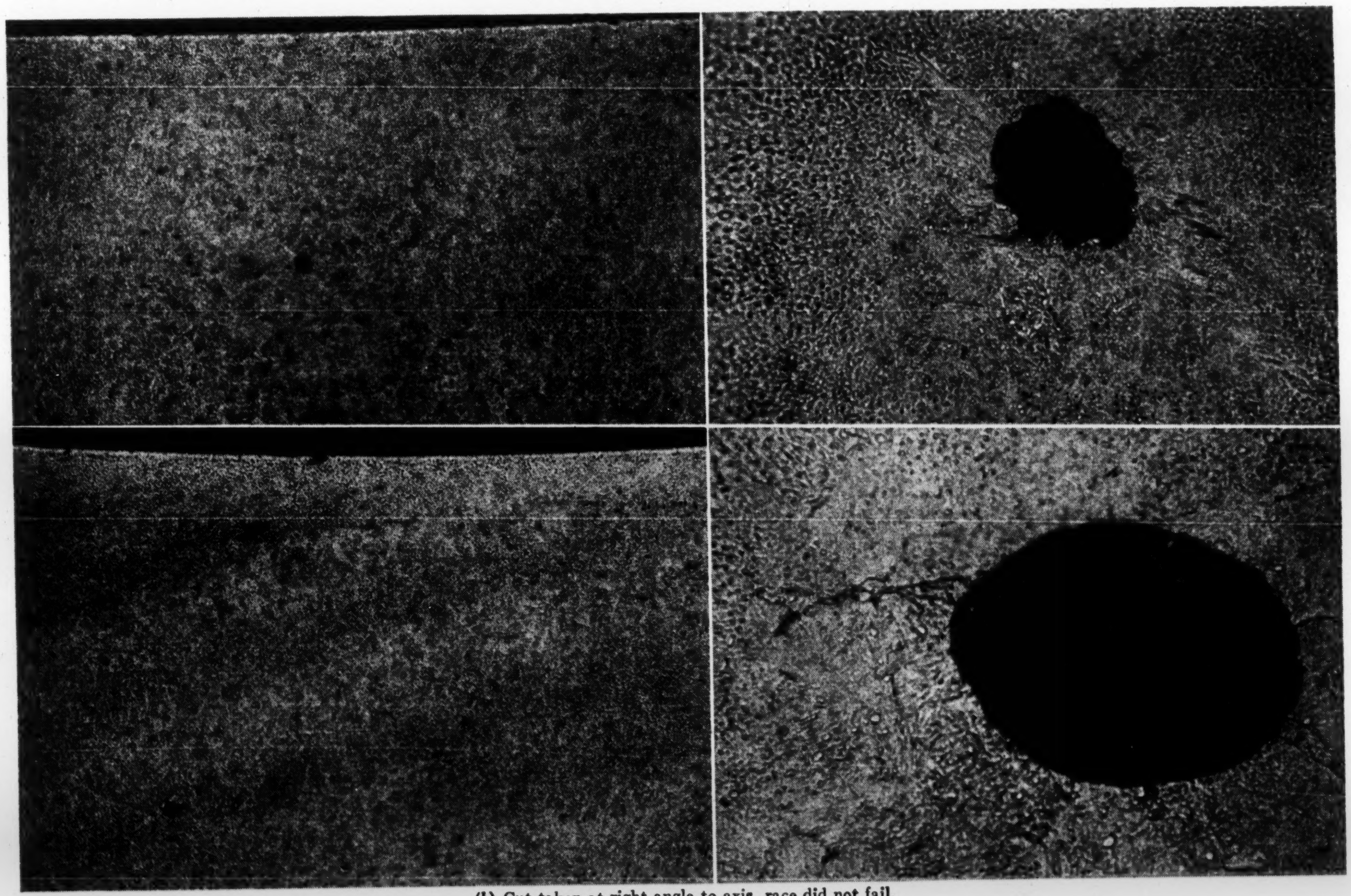
It is a tedious job to hunt for such starting points, because they are so small and are located at different distances from the surfaces even when the applied loads are as uniform as those in ball bearings which are subjected to pure thrust loads. Examples of the start of failures are shown in the excellent photomicrographs taken by H. O. Walp. Cuts taken at right angles to the axis are shown in Fig. 10(*a*), (*b*) and (*c*) and a radial cut parallel to the axis in Fig. 10(*d*). In Fig. 10 (*a*), (*c*), and (*d*) severe strain lines extend from slag inclusions at depths below the surface up to 0.03 in. It is of interest to note that the strain lines are not in any definite directional relation to the surface; some are at about 45 deg; others parallel to it. Figure 10(*b*) shows a crack without the presence of slag inclusions, essentially parallel to the surface, and 0.008 in. below. Figure 11(*a*) and (*b*) show start of cracks at slag inclusions. Figures 10(*a*) and 11(*b*) are cuts of races that did not fail. All photomicrographs at 100× show surface of races at ball path.

It is also very interesting that these severe strain lines often occur at a level below the dark etching region which has been subjected to heavy coldworking.

It is well known that many fatigue failures start from slag inclusions of various sizes, and such inclusions are therefore considered to be at least contributory to failures (13). Obviously they are dangerous only when they are located in a spot which is subjected to relatively heavy alternating shear stresses or tensile stresses. The results from rotating bending fatigue tests on vacuum-melted steel strongly indicate that defects other than slag inclusions are important. Local weak points may be due to crystal structure, segregation, and particularly to microstresses caused by the austenite to martensite transformation in hardening.

CONCLUSION

The life of ball bearings tested under the same conditions shows a wide scatter in a proportion of 1 to 50 or even more, (*a*) Cut taken at right angles to axis.



(*b*) Cut taken at right angle to axis, race did not fail.

×100 ×1000

FIG. 11.—Start of Cracks at Slag Inclusions.

and the individual failures generally start from elementary weak points that lie in or near the region of the greatest shear stress.

Fatigue tests on simple specimens of ring-type, rotating-beam-type, Krouse-type, and torsion-type specimens have shown that various careful heat treatments which give a hardness $\geqq$ 60 Rockwell C have little effect on the scatter in fatigue life or on the level of the load-life curve. With these specimens the surfaces can be finished to any degree required to avoid influence on the failure.

The median lives vary inversely to the applied maximum stresses about as follows: for the ring type as the twelfth power, for the necked-down rotating beam as the fifteenth, and for the torsion test as the tenth power. The median lives of ball bearings vary inversely as about the ninth or tenth power of the mean pressures at the contact point of rolling elements, suggesting that the failures in ball bearings start principally in the region below the contacting surfaces, where the highest shear stresses are located.

Elimination of certain obvious defects such as larger slag inclusions improves the fatigue life without reduction of the scatter.

Other local defects are clearly present to cause the wide scatter in life of heat-treated steel under changing stress. If reduction of local weaknesses can be obtained, a substantial increase in life of ball bearings should be possible.

Further tests on vacuum-melted steel are under way.

Acknowledgment:

It is a pleasure to acknowledge the great help and interested cooperation of my associates in the SKF Research Laboratory, which has made this presentation possible.

The tests which are recorded, and a great many more, have been run during the past 25 yr. The late F. W. Titus supervised the mechanical testing, and H. O. Walp the heat treating and metallographic work. Five years ago T. Morrison developed the pneumatic torsion tester under license agreement with the General Electric Co., and J. Porter and K. Clewell have collected the experimental data and drawn the diagrams.

REFERENCES

(1) K. Heindlhofer and H. Sjovall, "Endurance Test Data and Their Interpretation," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 45, p. 141 (1923).

(2) H. Styri, "General Properties of Ball Bearings", *Transactions*, Am. Soc. Mechanical Engrs., Vol. 47, p. 490 (1925).

(3) A. Palmgren, "The Carrying Capacity and Life of Ball Bearings," *The Ball Bearing Journal*, A/B Svenska Kullagerfabriken, Gothenburg, Vol. 3, p. 34 (1937).

(4) W. Jurgensmeyer, "Die Walzlager," Julius Springer, Berlin (1937).

(5) R. K. Allan, "Rolling Bearings," Sir Isaac Pitman & Sons, Ltd., London (1945).

(6) A. Palmgren, "Ball and Roller Bearing Engineering," SKF Industries, Inc., Philadelphia, Pa. (1945).

(7) H. Styri, "Fatigue Testing with Simple Test Specimens," *Metals and Alloys*, Vol. 4, September, 1933, p. 141.

(8) L. P. Tarasov and H. J. Grover, "Effects of Grinding and other Finishing Processes on the Fatigue Strength of Hardened Steel," *Proceedings*, Am. Soc. Testing Mats., Vol. 50, p. 668 (1950).

(9) J. O. Almen "Surface Deterioration of Gear Teeth," *Mechanical Wear*, Am. Soc. Metals (edited by Burwell), p. 239 (1950).

(10) F. B. Quinlan, "Pneumatic Fatigue Machine," *Proceedings*, Am. Soc. Testing Mats., Vol. 46, p. 846 (1946).

(11) H. B. Jones, "Metallographic Observations of Ball Bearing Fatigue Phenomena," *Proceedings*, Am. Soc. Testing Mats., Vol. 46, p. 853 (1946)..

(12) G. Lundberg and A. Palmgren, "Dynamic Capacity of Rolling Bearings," *Paper No. 48, Applied Mechanics*, Am. Soc. Mechanical Engrs., p. A-19 (1948).

(13) H. Styri "Remarks to Proceedings on Friction and Surface Finish," M. I. T. p. 182, (1940).

(14) "Evaluating Load Ratings of Annular Ball-bearings," Antifriction Bearing Manufacturers Association *Product Engineering*, December 1949.

(15) T. J. Dolan and C. S. Yen, "Some Aspects of the Effect of Metallurgical Structure on Fatigue Strength and Notch-sensitivity Steel," *Proceedings*, Am. Soc. Testing Mats., Vol. 48, p. 664 (1948).

DISCUSSION

MR. O. J. HORGER.[1]—I am certain that this paper by Mr. Styri has supplemented our knowledge of fatigue failures initiating from contact stresses. This problem has also been under investigation in our laboratory for many years as well as in others, and a clear understanding of contact stresses is still being sought.

In making fatigue tests of bearings or gear teeth the question always arises as to what degree of spalled condition of the surface is defined as a fatigue failure. There has been an increasing tendency toward requiring some arbitrary total area of spalled surface to develop in the test before failure is defined. A number of pin point spalls may develop at various stages in the life of a part. The development of the first pin point spall is not believed a good criterion of failure. Some of these pin point spalls may progress to larger spalled areas while others may show little progress. Pin point spalls which show little increase in size are not detrimental to the service performance of the part whereas the progressive type affect durability and may produce an objectional noise level. Scatter cannot be isolated unless a certain degree of spalling is established as a criterion of failure.

It is interesting to note that the *S-N* curve in Fig. 1 does not flatten off even at 1000 million revolutions, which appears to be characteristic of contact stresses. It is presumed in these tests of Fig. 1 that the inner race rotates while the outer race is stationary. The contact stresses on the inner race are higher than those on the outer race, but on the other hand more repetitions of stress are applied to the same point (in the region of the maximum ball load) on the outer race than on the inner. It would appear that the outer race would also present a spalled condition and often before the inner race. Possibly the author can present some comments on this question. Since the author discusses outer race failures in Figs. 2, 3, and 4 could he advise if the outer race is rotating or stationary?

Due to the large scatter in Fig. 1 the question is raised as to the influence of geometry variations in the bearings on bearing life. The deflections of the balls and contact areas are a very small thing and may be a large percentage of the manufacturing tolerances, even though these tolerances are also small. Does the author have any figures which could be submitted regarding the relationship of deflections and tolerances of the bearings investigated in Fig. 1?

The author shows how the slope of the *S-N* curve from the torsion tests approaches that for the bearing tests whereas the bending tests show a different slope. It is suggested that the torsion tests will better find weak points in the material than bending tests because equal shearing stresses act on both the longitudinal and transverse axis of the specimen and this is not the case in bending. For example, inclusions or grain direction due to processing operations may have an orientation more affected by shearing stresses in an axial direction produced by torsion (none in bending) than by shearing or tensile stresses on other planes obtained in either bending or torsion. The bearing tests represent contact stresses resulting in shearing stresses at the surface or below the surface more comparable with the direction-seeking characteristics for locating material weakness exhibited by the torsion tests. Does the author believe this explanation may be a reason for the equal slopes found in his tests?

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[1] Chief Engineer, Railway Division, The Timken Roller Bearing Co., Canton, Ohio.

It is worth while noting that many *S-N* curves in bending and torsion for these hard steels (Figs. 7, 8, and 9) shows (*a*) no definite flat portion at the customary ten million cycles and (*b*) considerable scatter at the lower part of the curve when a number of points are present (even though the author feels the scatter is less in torsion than bending). There are so few data of this type available that I would like to ask if the author considers very hard materials to characteristically exhibit a flat portion at a greater number of cycles and scatter to be much greater than found for engineering steels?

It would help if the author gave the critical dimensions of his fatigue specimens, and would comment as to whether the inclusions which incited failure were of the deoxidation or refractory type.

MR. J. M. LESSELLS.[2]—I am interested in the same question Mr. Horger has raised; namely, what was the criterion of failure?

We have done work in the past, using the Buckingham machine, which consists of two simple rollers in contact and eliminates the complication of a roller bearing. It was found that a large amount of scatter was obtained, similar to the results of Mr. Styri.

Another point refers to the possible application of theoretical reasoning to explain the results of subsurface failures. Based on the analytical work of Timoshenko, the maximum shear stress occurs at a certain depth below the surface, and this theory may be used as a check on the experimental data.

Since very little experimental information is available on the fatigue of surfaces this paper is of some considerable importance.

Incidentally, no reference is made by the author to data which have been published by Mr. Buckingham on surface fatigue.

CHAIRMAN T. J. DOLAN.[3]—I was very much impressed, with the evidence which Mr. Styri has shown of subsurface failures. There has been a controversy for some years as to whether fatigue failure begins from the surface or initiates as a subsurface phenomenon. I believe that he has offered some very convincing evidence on this subject.

I think the data showing scatter are perfectly normal and that they could be expected to show scatter.

MR. HAAKON STYRI (*author*).—In reply to Mr. Horger, we call it a failure when we can detect a noise change of the bearing during testing. The trained personnel we have in the laboratory can detect these failures at very small spalls which may be no larger than a pinhead.

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[2] Associate Professor of Mechanical Engineering, Massachusetts Institute of Technology, Cambridge, Mass.

[3] Research Professor of Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

MR. HORGER.—Do you use a special instrument for measuring?

MR. STYRI.—We use a "listening" stick, which is a simple kind of stethoscope, to observe the noise, and attendants can often detect the change in noise even when they sit in the office adjacent to the testing room because of the damping out of other types of noises. When they hear these bearings go bad, they stop the machine and take the bearing out for disassembly to examine the surfaces. Sometimes it may take up to fifteen minutes to find the spall by turning the balls and races around. The first observed spall is what we define as a failure of the bearing, because in these tests we are interested to learn how failures start.

In ball bearings such early failures usually progress rather rapidly under the test loads used. When roller bearings are tested it happens much more often that such spalls do not progress, but that more damaging spalls develop at another point. Obviously we should get a more uniform test with larger volume under stress, but also probably a lower life.[4]

We have wide experience in the effect of variation in geometry of the bearings, but due to the many factors involved, I cannot give figures that would satisfactorily cover all conditions. A great deal of experimental work remains to be done.

As to the flattening of the curve for bearing lives at lighter loads, we have not discovered any fatigue limit. We are running tests at very light loads at the present time and have run bearings up to 2.5 billion revolutions without getting failures. Still, we expect to get failure at this load. Maybe we will find some next year. It is possible that there is a fatigue limit at which bearings will not fail, but I think that depends on whether or not we have weak spots in the material in the neighborhood of the ball track. As for flattening of S-N curves for simple specimens this is commented on under joint discussion.

The lives recorded for both types of bearings tested are based on number of inner race revolutions. For the No. 6207 one row groove bearings, only inner race lives are given; for the No. 1207 two row self-aligning ball-bearing, only outer race lives. In both cases the lives are given for the part of the bearing that has the great majority of failures. Other parts do fail, but usually to a much lesser extent, and are not included in this investigation.

The question was asked as to whether the causes of failures are similar to those in torsion-type specimens. We have shown that the slope of the fatigue curve in the torsion-type specimen is about the same as we have found for bearings when we use the computed normal contact stresses as the load *versus* the life, while, when we use the computed bending stresses as we have in the rotating ring, or in the rotating-beam type for load stress *versus* life, the slope of the fatigue curve differs.

Weak points in material may be found in reversed bending tests and with reversed torsion tests. It was shown in a previous discussion, on the paper by Tarasov (8), that reversed bending of specimens with stress transverse to direction of fiber gave lower lives than for stress lengthwise to the fiber. The rotating ring is also subject to reversed stresses transverse to the fiber direction while the rotating cantilever pins have reversed stresses parallel to the fiber direction.

In addition to that, we have the well-known fact that the shear stresses some distance below the surfaces of contact in gears and bearings are higher than at the surface, and these shear stresses vary in direction with the passing of the rolling element. Etching of a section also indicates that we have disturbed material some few thousandths below the contacting surface. If we then have impurities or other weak points in the material, in that range where we have high shear stresses, they may produce nuclei for the failures.

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[4] W. Weibull, "A Statistical Theory of the Strength of Materials," *Proceedings*, Royal Swedish Inst. for Engineering Research, Nr. 151 (1939).

It is of interest, in this connection, that when we test the torsion-type hardened specimens, we always get the first indication of failure in the direction of the axis or parallel with the fiber, and not transverse. From both ends of this longitudinal crack the failure spreads out at about 45 deg. in one direction or the other.

In answer to Mr. Lessells question whether subsurface failures always start, or predominantly start, where the maximum shear stresses are present, I am not so sure that they do. As a matter of fact, we very often see that the nuclei for cracks are either below the region of the maximum shear stresses, or at some other place a little away from this region (see Fig. 4), and these nuclei are often slag inclusions of the oxidic type, but may be other weak points. If we had no defect in the range where we have the maximum shear stresses, I would say we most likely would have failure starting there, and the crack shown in Fig. 10(*a*) is actually found about where the maximum shear stress is, 0.006 in below the surface, as calculated from the load we have on the ball track at that point.

From our own observations, we think that approximately nine tenths of all the failures start below the surface. If we have a rough scratch or something like that on the surface, we may get the fatigue failure starting there, but that is unusual.

Unfortunately, I have not come across any fatigue tests performed by Buckingham or others on high-carbon steel specimens heat treated to 60 Rockwell C or over, and very little of such material is available in published literature. Many other theoretical treatises, besides those already given, have, of course, been published.[5,6]

MR. HORGER.—Does the failure referred to as initiating below the surface and comments regarding surface finish pertain to tests on contact stress or the plain bending and torsion specimens?

MR. STYRI.—They refer to contact stresses. However, in the Krouse-type specimen, as well as in the torsion-type and rotating-beam type, we also often find the nuclei for the fatigue cracks at points below the surface of maximum stress. These plain specimens are finished very carefully on the surfaces so that we do not have any scratches that would be likely to affect the life. That is the very reason why we selected these for tests.

The dimensions of the specimens are:

Ring type:	
OD, in	2
Bore, in	1¾
Width, in	½
Cantilever beam:	
Neck diameter, mm	2.5
Torsion spec.:	
Diameter, mm	4
Length, mm	16

[See also Joint Discussion of Styri and Gross papers, p. 719—ED.]

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[5] E. Buckingham, "Analytical Mechanics of Gears," McGraw-Hill Book Co., Inc., New York, N. Y. (1949).

[6] Shaw and Macks, McGraw-Hill Book Co., Inc., New York, N. Y. (1949).

LABORATORY EVALUATION OF MATERIALS FOR MARINE PROPULSION GEARS*, [1]

BY M. R. GROSS[2]

SYNOPSIS

The purpose of this paper is to present the results of tests performed to evaluate the pitting resistance and root fatigue strength of materials for marine gear applications. By means of contact roller test machines, pitting limits have been determined for six steels and eight non-ferrous materials. The breakdown limits for five plastic base materials have also been determined. A new test called the "simulated gear tooth fatigue test" has been developed to evaluate the root fatigue strength of materials under reversed bending stresses. The effect of hardening treatments, root radii, and surface finish and treatments on the endurance limit of the root area has been determined for three steels. Conclusions based on the results of the tests and relative ratings for the ferrous materials are presented.

Immediately after World War II, the Bureau of Ships of the Department of the Navy inaugurated a program to reduce the weight and space requirements of many shipboard components. Considerable attention was focused on the reduction in weight of propulsion gearing, and a great deal of effort has been and is being expended in this direction. New and novel high-speed, lightweight propulsion gears have been designed and constructed by the foremost marine gear manufacturers in the country. In addition, the Navy has set up extensive facilities for the testing of full-scale experimental gears to destruction.

Early in the program it was realized that such large scale destructive tests would be expensive and could be conducted on a limited number of gears. Accordingly, it was decided to conduct relatively inexpensive laboratory tests in order to evaluate various materials and treatments and, if possible, to correlate these small-scale tests with the full-scale destructive tests. It is the purpose of this paper to describe the types of laboratory tests which were performed and to present the results obtained.

A review of naval gear failures at the time the laboratory tests were initiated showed the majority of failures to be caused by (*a*) pitting of the tooth contact surfaces because of cyclic compressive stresses and (*b*) breaking of the teeth in the root areas because of cyclic bending stresses. Accordingly, it was decided that laboratory tests should simulate conditions responsible for both types of failures. Way[3] Buckingham,[4] Knowlton and Snyder,[5] and others describe roller tests which simulate surface stress conditions existing at the pitch line of the gear. Because of its simplicity, Way's

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[*] Presented at the Fifty-fourth Annual Meeting of the Society, June 18–22, 1951.

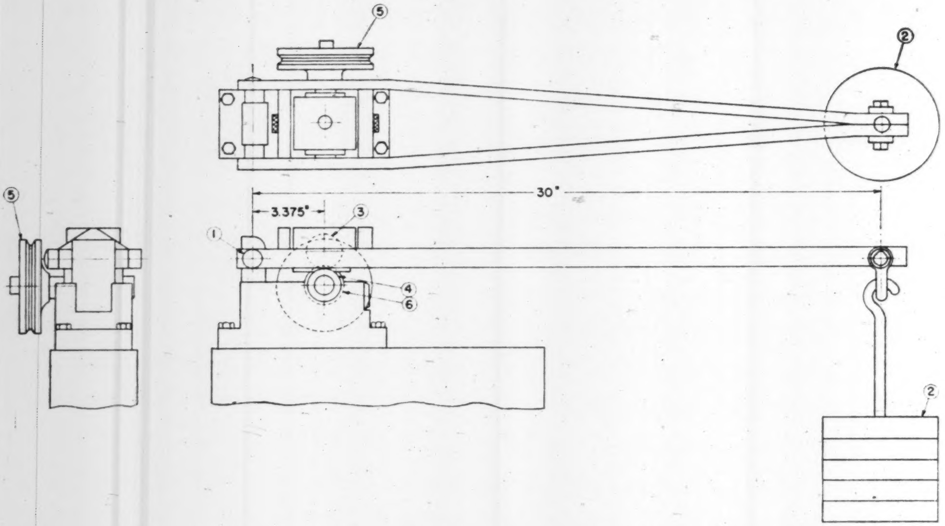
[1] The views presented in this paper are those of the author and do not represent necessarily the opinions of the Navy Department.

[2] Metallurgist, U. S. Naval Engineering Experiment Station, Annapolis, Md.

[3] S. Way, "Pitting Due to Rolling Contact," *Journal of Applied Mechanics*, June, 1935, pp. A49–A58.

[4] E. Buckingham, "Qualitative Analysis of Wear," *Mechanical Engineering*, August, 1937, p. 576.

[5] H. B. Knowlton and E. H. Snyder, "Selection of Steel and Heat Treatment for Spur Gears," *Transactions*, Am. Soc. Metals, Vol. 28, pp. 687–708 (1940).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30"
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FIG. 1.—Details of Testing Machine for Subjecting Contact Rollers to Compressive Stress.

1. Pivot
2. Weights added to beam and hanger
3. Contact test roller
4. Drive roller
5. Pulley on driving shaft
6. Ball bearing

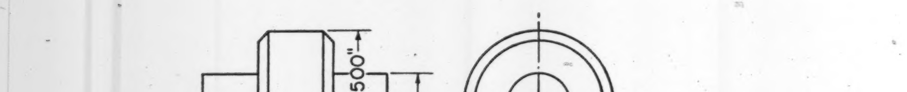
1.500"
0.570±0.002
0.790"-0.791"
1 7/8"
Upper Roller
Press Fit for Ball
Bearing No. 303
3/32" Rad
1.562"
1"/8
0.662"
7"/8
47"/64
17"/32
47"/64
5"/8
2 1/2"
6"
Lower Roller

FIG. 2.—Contact Roller Specimens.

contact roller apparatus was selected for this investigation. To simulate the flexural fatigue failure of the gear teeth in the root areas, a new test was developed. This has been called the "simulated gear tooth fatigue test."

Contact roller tests have been conducted on a wide variety of ferrous, nonferrous, and non-metallic materials. The incorporation of some of the light metals and plastic base materials in the test program may seem odd. However, the Navy has certain low-power gear applications where quiet operation is an important factor, and it was requested that certain high damping materials be tested. The results of these tests are presented because of the general interest in many of these materials.

Only ferrous materials have been investigated in the simulated gear tooth fatigue test. Tests on variations in root finish and radii together with various hardening and surface treatments are included in the text.

CONTACT ROLLER TEST

A drawing of the test machine which simulates the rolling action that occurs at the pitch line of a gear tooth is shown in Fig. 1. The test machine is self-aligning and requires two cylindrical rollers similar to those shown in Fig. 2. The lower and upper rollers rotate together along parallel axes while being subjected to a compressive load. Both rollers are supported by ball bearings, and the lower roller is driven by a pulley attached to the projecting end. The upper roller is driven by traction, and no intentional slippage is introduced between the rollers. The upper roller is considered to be the test roller in all instances; and, unless otherwise noted, the lower roller is carbon tool steel hardened and tempered to 63 Rockwell C. The speed of the lower roller is maintained at approximately 700 rpm and this roller dips in a bath of Navy Symbol 2190T oil. Sufficient oil is picked up to provide an ample supply of lubricant at the contact surfaces.

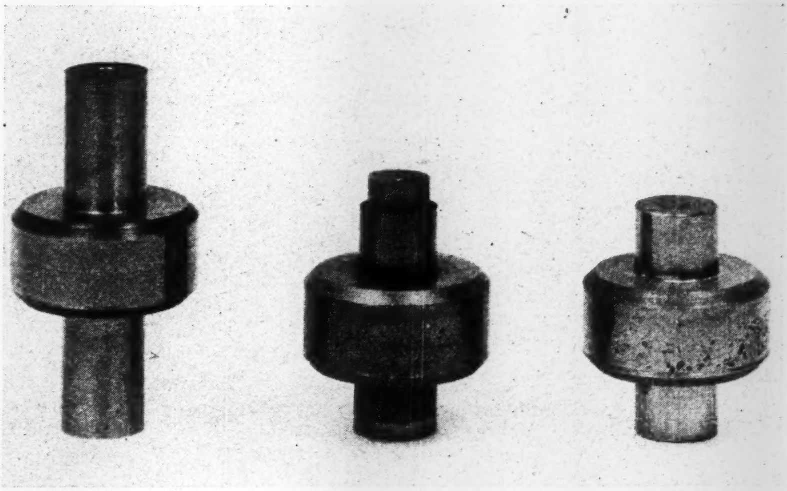

FIG. 3.—Pitted Contact Rollers.

Prior to the presentation of contact roller test results, it is desirable to define certain descriptive terms used in this paper. It was recognized at the outset that, in order to define a contact roller failure, it would be necessary to specify the minimum size and number of pits.

In order to be consistent with work done previously by other investigators, a failing pit is described as one having an area of 0.0001 sq in. or greater as measured on the contact surface. A failed specimen is further defined as one having at least one failing pit per square inch of contact surface. In this investigation, the appearance of three or more pits 0.0001 sq in. in area or greater on any one roller constituted a failure.

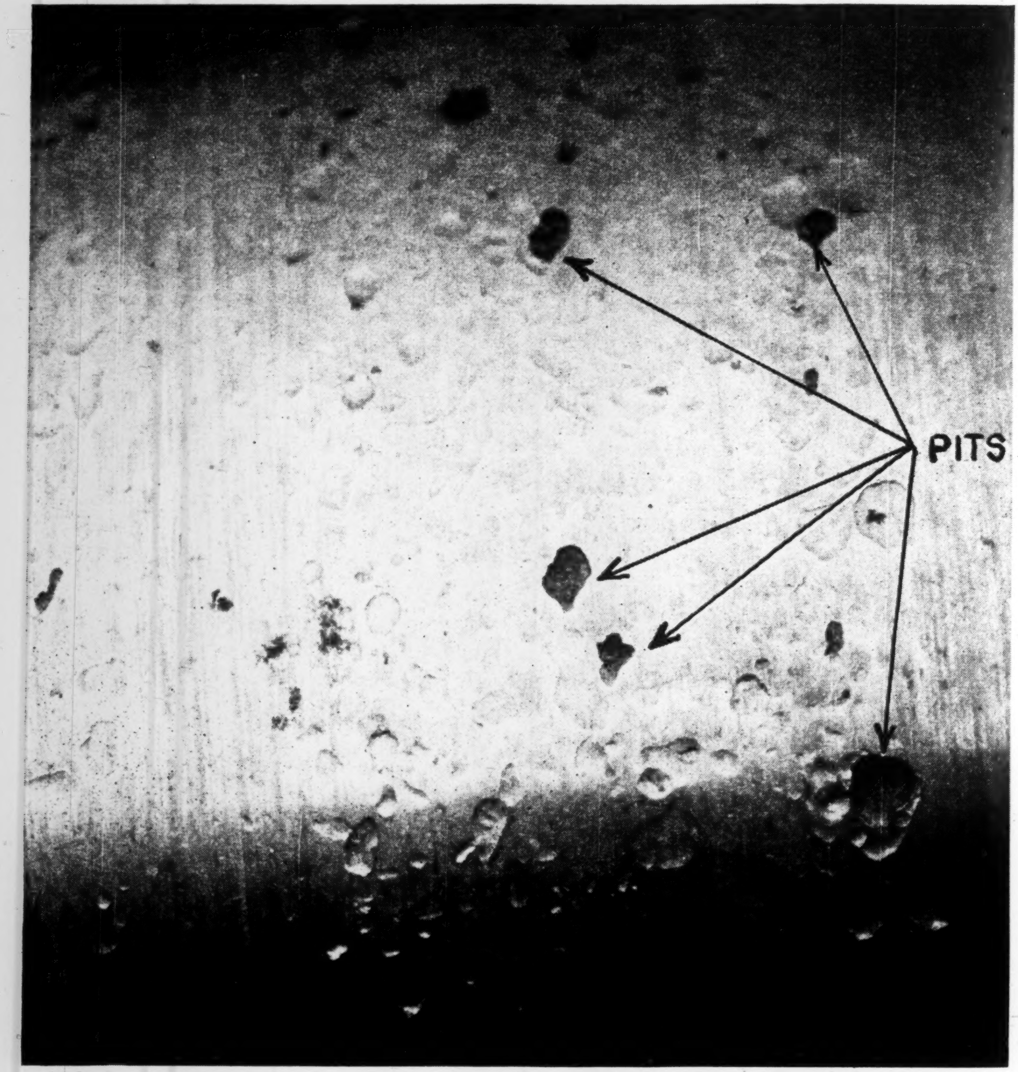

FIG. 4.—Contact Roller Surface Showing Typical Pits (×12).

The "theoretical maximum compressive stress," $P_{\max}$ is defined as the maximum unit pressure at the middle of the rectangle of contact formed by two rollers in compression. In the nineteenth

Century, Hertz mathematically defined P_{max} and his equations for two parallel cylinders can be found in strength of materials textbooks.[6, 7]

There is little doubt that pitting is the result of fatigue failure of the metal, the initial crack starting at or immediately below the surface and then being propagated by repeated stresses until a particle of metal is expelled and a pit is observed. Exactly how the initial crack forms is not readily apparent, but it does so only after many thousands or millions of cycles. In many instances, the cyclic manner of failure responsible for pit formation results in the "oyster shell" appearance of the fractured areas. This is peculiar to progressive fractures produced by fatigue. Typical contact roller pits are shown in Figs. 3 and 4.

In addition, the development of so-called stress-cycle curves from the contact roller tests reported herein and elsewhere supports the viewpoint that pitting is a fatigue phenomenon. For example, if a series of rollers made from the same ferrous material are subjected to different stress levels, the number of cycles to failure is related to the stress, as shown in Fig. 5. These curves are identical in shape to those found in ordinary flexural fatigue testing. In the contact roller test, the so-called "pitting limit" corresponds to endurance limit of the ordinary fatigue test. In this paper, the "pitting limit" is defined as the maximum compressive stress below which a specimen will not fail in less than 10,000,000 cycles.

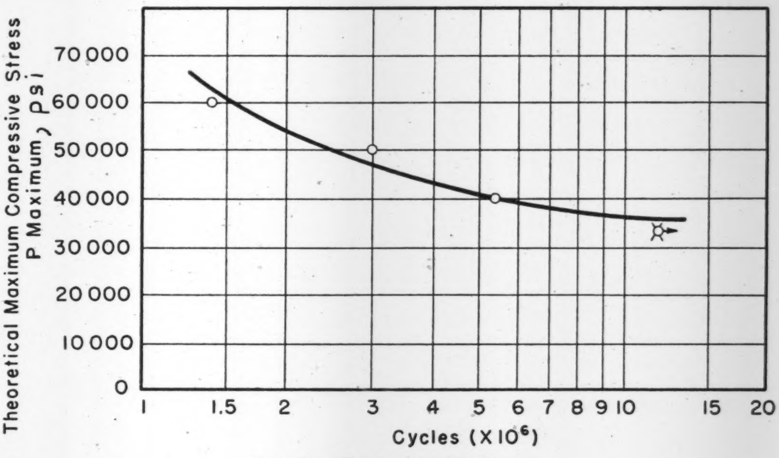

FIG. 5.—Contact Roller Test.

Ferrous Materials:

Contact roller tests have been conducted on the following ferrous materials:

Steel A—SAE 1035 steel
Steel B—2½ per cent Ni, ½ per cent Mo steel
Steel C—1 per cent Cr, 2½ per cent Ni, ½ per cent Mo, ¼ per cent V steel
Steel D—5 per cent Cr, ½ per cent Mo steel
Steel E—NE 8615 steel
Steel F—SAE 4615 steel

Steels A and B have been specified by the Navy for many propulsion gears. Obviously the properties of these materials were fully investigated in order to form a basis of comparison with other

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[6] R. J. Roark, "Formulas for Stress and Strain," McGraw-Hill Co., New York, N. Y., (1943).

[7] S. Timoshenko, "Strength of Materials—Part II," Van Nostrand Co., New York, N. Y., Sec. Ed. (1941).

TABLE I.—SUMMARY OF CONTACT ROLLER TEST DATA FOR FERROUS MATERIALS.

Steel	Type	Source	Treatment	Chemical Composition, per cent							Tensile Properties					Brinell Hardness Number	Pitting Properties		Rating
				Carbon	Manganese	Silicon	Chromium	Nickel	Molybdenum	Vanadium	Tensile Strength, psi	Yield Strength, 0.2 per cent offset, psi	Proof Stress, psi	Elongation in 2 in., per cent	Reduction of Area, per cent		Pitting Limit psi	Load-Carrying Capacity, lb per lin in.	
A	SAE-1035	Destroyer escort bull gear	As rec'd	0.36	0.71	0.29	0.35	Trace	0.00	0.01	81 200	47 500		34	66	153	36 000	95	1.0
B	Ni-Mo	Aircraft carrier pinion gear	As rec'd	0.33	0.63	0.28	0.09	2.70	0.44		102 400	67 500	61 300	25	55	213	63 000	290	3.1
B	Ni-Mo	Aircraft carrier pinion gear	Quenched and drawn at 800 F	0.33	0.63	0.28	0.09	2.70	0.44		186 000	167 200	162 500	10	33	352	84 000	515	5.4
B	Ni-Mo	Aircraft carrier pinion gear	Quenched and drawn at 600 F	0.33	0.63	0.28	0.09	2.70	0.44		216 000	184 000	176 200	7	14	432	122 000	1090	11.5
B	Ni-Mo	Aircraft carrier pinion gear	Quenched and drawn at 400 F	0.33	0.63	0.28	0.09	2.70	0.44		244 000	188 000	156 200	10	29	490	142 000	1480	15.6
C	Cr-Ni-Mo-V	18 in. Diam. Forging	Normalized and drawn	0.24	0.72	0.17	1.07	2.37	0.47	0.22	134 500	116 800	101 500	19	53	283	60 000	265	2.8
C	Cr-Ni-Mo-V	18 in. Diam. Forging	Nitrided 100 hr at 975 F	0.24	0.72	0.17	1.07	2.37	0.47	0.22						Surface 560	>250 000	>4580	>48.3
D	Cr-Mo	18 in. Diam. forging	Normalized and drawn	0.26	0.47	0.24	4.75	0.19	0.48		124 800	102 700	92 100	20	60	270	66 000	320	3.4
E	NE-8615	1$\frac{15}{16}$ in. diam. bar	Case carburized	0.17	0.79	0.25	0.52	0.53	0.17							Surface 600	>250 000	>4580	>48.3
F	SAE-4615	1¾ in. diam. bar	Eutectoid carburized	0.16	0.53	0.31	0.24	1.64	0.27							Surface 710	>250 000	>4580	>48.3
F	SAE-4615	1¾ in. diam. bar	Carbonitrided	0.16	0.53	0.31	0.24	1.64	0.27							Surface 720	>250 000	>4580	>48.3

materials and treatments. Steels C and D have previously been investigated to ascertain their suitability for turbine rotors. When received, these two steels had been heat treated so as to attain high strength in large sections, without liquid quenching, and as such were considered worthy for investigation as gear materials. Steels E and F were used to determine the effects of carburizing treatments.

Figure 5 is a typical stress-cycle curve for ferrous materials. Curves such as these were established and the pitting limits determined for steels A through D, inclusive, both in the as-received and treated conditions. Steel E was carburized and steel F was both eutectoid carburized and carbonitrided. Tests on the case-hardened steels were conducted at a stress of 250,000 psi, the maximum stress which can be applied with the present facilities, and pitting did not occur. In these latter tests, both the upper and lower rollers were identical in composition and treatment. This was also the case for steel C which had been nitrided for 100 hr at 975 F. The source, chemical composition, treatment, mechanical properties, hardness, pitting limit, and load-carrying capacity of the ferrous materials tested are tabulated in Table I. The "load-carrying capacity" is given in pounds per lineal inch and is defined by the compressive load on the rollers in pounds, necessary to produce the stress value shown for the pitting limit, divided by the length of the contact surface. This provides a convenient means for comparing all types of materials since the moduli of elasticity and Poisson's ratios are not included as in the Hertz equations. It is to be noted in Table I that the materials have been given a rating which is dependent upon their load-carrying capacity. Steel A, a steel used in many marine gear applications, has arbitrarily been given a rating of 1.0. The large increase in load-carrying capacity resulting from the case-hardening treatments is readily apparent.

In addition to the aforementioned tests, induction-hardened and chromium-plated (0.005 in. chromium) rollers of steel B were tested at maximum compressive stresses of 150,000 psi and 200,000 psi, respectively. The induction-hardened roller behaved similarly to the quenched-and-drawn specimens of high hardness, and the chromium-plated rollers readily spalled.

It is customary in some design practices to assume a linear relationship between resistance to pitting and Brinell hardness. Figure 6 is a plot of the data for the throughly hardened steels shown in Table I. The linearity of the relationship adds credence to the assumption. It should be recognized, however, that extrapolation of this relationship fails to predict the results obtained for the nitriding and carburizing treatments. This is as would be expected, however, since exceptionally high pitting limits for these treatments undoubtedly result from the compressive stresses inherent in the case.

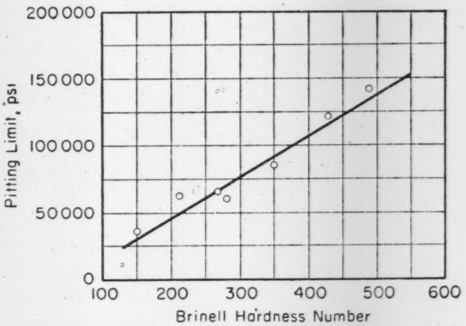


FIG. 6.—Hardness *versus* Pitting Limit for Through Hardened Steels.

During the course of the investigation, it was noted that many of the rollers, under certain conditions of loading, showed extreme surface wear. This was accompanied by a decrease in diameter and a discoloration and contamination of the oil bath. Examination of these surfaces at low magnification revealed that the wear was due to deterioration of the surface caused by fine general pitting. Such a surface is shown in Fig. 7. At the conclusion of the test, the specimens were separated into two categories as to those which showed general pitting and those which did not. The results of this separation for steel B is plotted in Fig. 8.

The rapid wear of these rollers is of interest since it appears that a limit is reached beyond which wear is exceedingly rapid regardless of hardness. Actually, the maximum pitting limit of which this alloy is capable is considerably impaired by the interposition of wear.

The effect of surface finish on the pitting resistance of gears has been a matter of discussion for many years. With this in mind, a series of contact roller tests was conducted to determine the effects

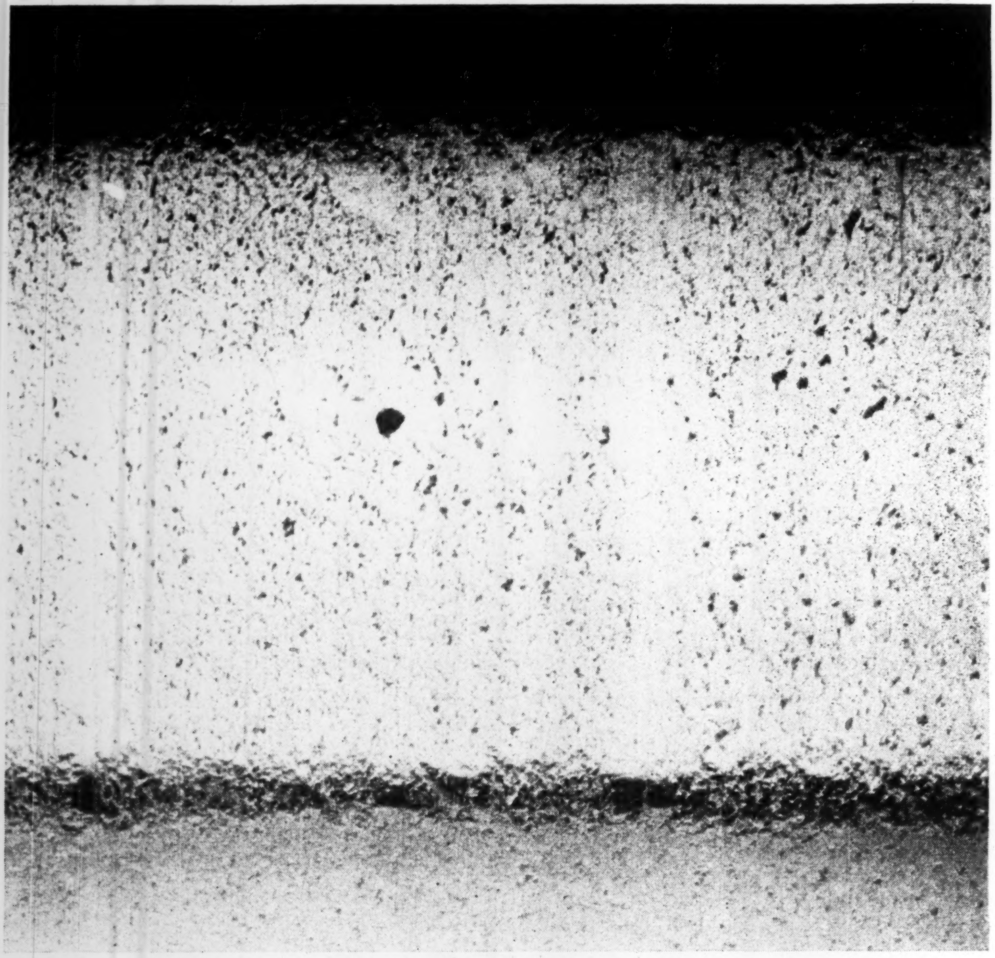


FIG. 7.—Contact Roller Surface Showing General Pitting (×12).

of various surface finishes on the pitting resistance of the as-received steel B. Both the upper and lower rollers were of the same hardness and had the same surface finish. Based on previous experience, a theoretical maximum compressive stress of 80,000 psi (470 lb per lineal in.) was selected as the most suitable stress level for differentiating between the various surface finishes. Fortunately, this selection proved most satisfactory and provided the data shown in Table II. The finishes are listed in order of their increasing resistance to pitting. The superiority of the fine-ground finish is readily apparent.

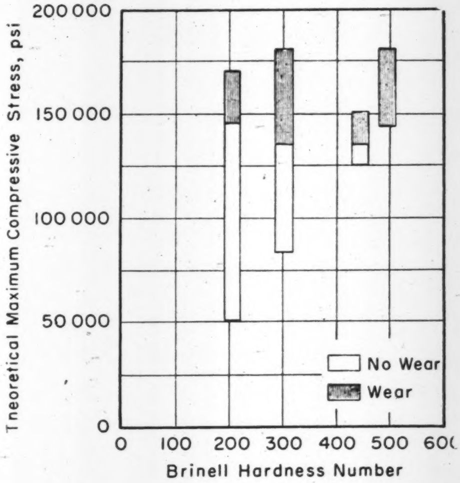


FIG. 8.—Graph Showing Conditions Under Which Steel B Exhibited Wear in Contact Roller Tests.

The tests on surface finish just described have been made on one material at one hardness level. Consequently, it is not certain how the various finishes would respond when applied to a material of higher hardness. The primary purpose of adopting a higher hardness level is to take advantage of an increase in load-carrying capacity. It is well known that the fatigue behavior of steels becomes more sensitive to surface conditions as the hardness increases. Accordingly, it would appear that the surface finish would become increasingly important at higher hardness levels.

Non-ferrous Materials:

The pitting properties have been determined for the non-ferrous materials listed in Table III. Upper contact rollers prepared from the compositions shown were run against lower rollers which were made of either hardened-and-tempered tool steel or an alloy identical in composition to the upper roller. Because of their low modulus of elasticity, non-ferrous lower rollers were used to obtain the lower stress values for the aluminum, magnesium, and manganese-copper compositions.

TABLE II.—EFFECT OF SURFACE FINISH ON THE PITTING PROPERTIES OF STEEL B.
(As-received hardness-213 Brinell Hardness Number)

Surface Finish	Theoretical Maximum Comp. Stress, psi	Cycles To Failure
		$\times 10^6$
Vapor Blast—140 QVB	80 000	0.3
Shot Peened—P33-0.015A-2	80 000	0.3
Shot Peened—P33-0.015A-2	80 000	0.6
Vapor Blast—1250 NVB	80 000	1.1
Parco Lubrite	80 000	0.5
Parco Lubrite	80 000	1.1
Ground—50 rms	80 000	1.6
Ground—50 rms	80 000	1.6
Ground—18 rms	80 000	3.0
Ground—5 rms	80 000	10.4 (1 pit)
Ground—5 rms	80 000	10.5 (2 pits)

Scoring of the contact surfaces invariably resulted when rollers of identical composition were run together. In the case of the aluminum alloys, even the pairing of alloys having the greatest hardness differential, that is, alloys G and J, failed to prevent scoring.

The pitting limits of these alloys, based on the stress necessary to cause failure in 10 million cycles, are shown also in Table III. The type of pitting on the contact surfaces of these alloys was

TABLE III.—SUMMARY OF CONTACT ROLLER TEST DATA FOR NON-FERROUS MATERIALS.

Alloy	Type and Treatment	Source	Nominal Composition, per cent								Typical Tensile Properties			Brinell Hardness Number	Surface Roughness, microinch	Pitting Properties[b]	
			Copper	Silicon	Magnesium	Manganese	Aluminum	Zinc	Cerium[a]	Beryllium	Tensile Strength, psi	Yield Strength, psi	Elongation in 2 in., per cent			Pitting Limit, psi[c]	Load Carrying Capacity, lb per lin in.
G	Wrought Al 14S-T6	1¾ in. rd x 10 in bar	4.4	0.8	0.4	0.8	Rem				70 000	60 000	13.0	136	25	38 000	206
H	Sand-cast, Al 356-T4	1¾ in. rd x 10 in. bar		7.0	0.3		Rem				28 000	16 000	6.0	80	25	<20 000	<57
I	Sand-cast Al 319-T61	1¾ in. rd x 10 in. bar	3.5	6.0			Rem				41 000	36 000	1.0	124	25	24 000	91
J	Sand-cast Al 220-T4	1¾ in. rd x 10 in. bar			10.0		Rem				46 000	25 000	14.0	80	25	<20 000	<57
K	Cu-Be Prec. hard. ASTM-B-120	1¾ in. rd bar	Rem							2	Min 150 000	Min 85 000	Min 3.0	333	13	56 000	315
L	Sand-cast aged dow-metal-"C"	1¾ in. rd x 10 in. bar	0.04	0.07	Rem	0.18	8.6	1.9			39 700	21 300	2.0	74	47	27 000	150
M	Sand-cast aged dow-metal-"E6"	1¾ in. rd x 10 in. bar			Rem	0.07			5.2		18 200	12 500	0.2	49	47	<20 000	<82
N	Mn-Cu	1¾ in. rd x 7 in. bar	22			78								103	13	<15 000	<24

[a] Total rare earths

[b] Stress value based on failure at 10 million cycles

[c] Calculations based on the following moduli of elasticity:

Al	10.3×10^6 psi
Cu-Be	17×10^6 psi
Mg	6.5×10^6 psi
Mn-Cu	20×10^6 psi
Steel	30×10^6 psi

identical to that found in the ferrous alloys, and the same criteria for failure were used. However, the shape of the stress-cycle curve for the magnesium and aluminum alloys differed from that of steel in that no distinct leveling-off occurred within 10 million cycles. Therefore, the defining of the pitting limit for these alloys requires the specification of the cycle parameter. On the other hand, tests on hardened copper beryllium, alloy K, resulted in a stress-cycle curve similar to steel. The behavior of the aluminum and magnesium alloys is similar to that encountered in ordinary fatigue tests of non-ferrous materials, and further substantiates the theory that pitting is a fatigue phenomenon.

As for steel, the wear in these materials was accompanied by general pitting of the surface and contamination of the lubricant.

Non-metallic Materials:

The non-metallic materials tested are tabulated in Table IV. Material O is an unlaminated polyamide resin, whereas the balance of materials are laminated phenolic resins having a woven cotton fabric filler. Materials P and Q are identical except for the orientation of the filler laminae. The filler in P is more or less randomly distributed, whereas that in Q is oriented in parallel planes. Test specimens from the latter were machined so as to have the laminations perpendicular to the specimen axis. The weight of the filler used in materials P and Q is not known. On the other hand, the filler in materials R, S, and T is 10, 12, and 15 oz cotton fabric, respectively. The laminations in the latter three materials are oriented in the same direction as those in material Q.

Because of the heterogenous nature of all but one of the materials tested, the same criteria for failure as set forth for steel were not used. Instead, the number of cycles necessary to cause a breakdown of the surface under a given load or stress was determined. This breakdown was evidenced in two ways. First, by a pronounced vibration of the loading beam; and, second, by visible deterioration of the contact surfaces. In general, the breakdown occurred over localized areas and appeared to result from the disintegration of the plastic resin. In several instances the over-all diameter of the test roller was reduced. Although this phenomenon may be regarded as a form of wear, it is believed that it resulted from plastic deformation of the contact surfaces.

The first contact roller tests were performed on material P. As stated previously, the laminae in this material were randomly distributed and thus were deployed at various angles with the contact surfaces. Furthermore, specimens of identical material were run together. This particular combination of conditions, when tested at a stress of 22,000 psi, caused the contact surfaces to become extremely hot, and failure ensued in 3 min (2200 cycles). Failure resulted from the shearing of the laminae and further tests on this material were abandoned.

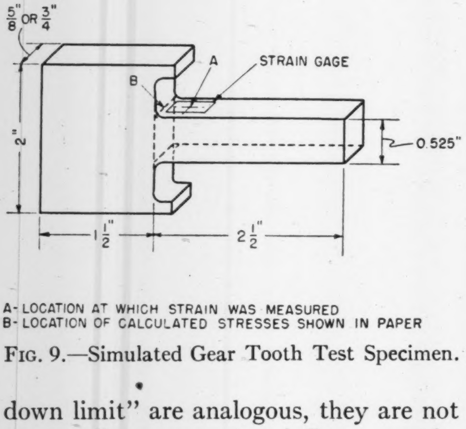
Upon examining the stress-cycle relationships for these materials it was noted that the curves evidenced a leveling-off at approximately 10 to 15 million cycles. It was possible, therefore, to determine a so-called "breakdown limit" which may be considered analogous to the "pitting limit" found in previous tests. Visual examination of rollers which did not fail within 10 million cycles showed no evidence of deterioration or deformation of the contact surfaces. The theoretical maximum compressive stresses and the load-carrying capacity at the estimated breakdown limit for the various materials are tabulated also in Table IV.

In connection with the foregoing paragraph, it is necessary to distinguish between the load-carrying capacities of the plastic-base materials and other materials tested in the investigation. This follows from the fact that although the terms "pitting limit" and "breakdown limit" are analogous, they are not necessarily synonymous. For example, "breakdown limit" indicates a stress or a load above which the material will break down within a few million cycles and thus produce a complete failure. On the other hand, the "pitting limit" indicates a degree of breakdown dependent on the test criteria and does not necessarily mean the material will not perform satisfactorily at higher stress or load levels. In fact, the size of pits arbitrarily set up as a test criterion for establishing the "pitting limit" would probably go unnoticed in normal propulsion gear operation.

TABLE IV.—SUMMARY OF CONTACT ROLLER TEST DATA FOR NON-METALLIC MATERIALS.

Material	Type	Filler	Breakdown Properties	
			Breakdown Limit, psi[a]	Load Carrying Capacity, lb per lin in.
O	Nylon—FM1	None	13 000	190
P	Molded Phenolic	Cotton fabric	Not determined	Not determined
Q	Laminated Phenolic	Cotton fabric	12 000	165
R	Laminated Phenolic	10 oz-cotton fabric	16 000	290
S	Laminated Phenolic	12 oz-cotton fabric	11 000	135
T	Laminated Phenolic	15 oz-cotton fabric	13 000	190

[a] Calculations based on moduli of elasticity of 1 × 10^6 psi and 30 × 10^6 psi for plastic-base material and steel, respectively.



A- LOCATION AT WHICH STRAIN WAS MEASURED
B- LOCATION OF CALCULATED STRESSES SHOWN IN PAPER

FIG. 9.—Simulated Gear Tooth Test Specimen.

The low thermal conductivity and the abrasive nature of the plastic-base materials should not be overlooked. The heat generated under certain conditions during the contact roller tests was not dissipated from the contact surfaces. This resulted in a softening and disintegration of the phenolic resin. The abrasive action of the phenolic materials on unhardened steel is well known. The fact that in many gear installations the cooling and lubricating oil systems are one and the same requires that suitable filters be installed in plastic gear systems to remove the abrasive particles in the event partial breakdown occurs.

SIMULATED GEAR TOOTH FATIGUE TESTS

As mentioned previously, a test was designed to simulate the bending fatigue stresses which occur in the root area of a gear tooth. Specimens for this test are $\frac{5}{8}$- or $\frac{3}{4}$-in. wide sections cut from a 5- to 9-in. long T-shaped test block. A typical specimen is shown in Fig. 9. The test blocks were prepared by a marine gear manufacturer, and the fillet areas were cut on a standard gear cutting machine. The specimens were sawed from the test block and the sawed surfaces ground parallel. The corners at the fillets were then rounded to minimize any so-called "edge effect."

The specimens were subjected to completely reversed cyclic bending stresses in Westinghouse Vibration Fatigue Equipment, Type HI-40. This apparatus operates at the resonant frequency of the mechanical system which includes the test specimen. During testing, the large end of the specimen is held stationary while the projecting end, representing the gear tooth, is vibrated up and down. Thus the projection behaves as a cantilever beam.

Throughout the vibration fatigue tests, the stress in the specimen was determined by means of an SR-4 resistance strain gage, type CD-7, cemented to the surface of the projection as shown in Fig. 9. In order to determine whether the maximum fiber stress at location *A*, as determined by the strain gage, agreed with that calculated from the beam formula, the specimen was statically loaded and the strains recorded at various loads. Results were in reasonably good agreement. It is emphasized, however, that the stresses presented in this paper for the gear tooth specimens are not those at location *A* but rather the maximum fiber stress at location *B*. Disregarding the change in section resulting from the fillets, location *B* is at the intersection of the projection with the root end of the specimens.

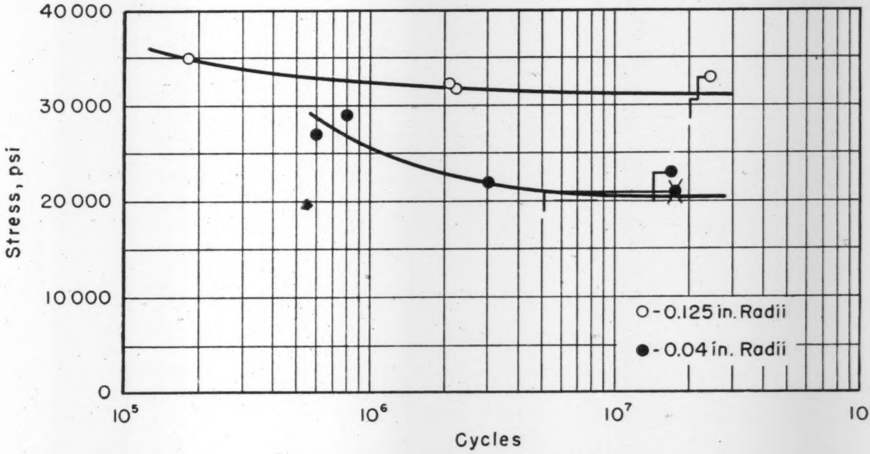


FIG 10.—Stress-Cycle Relationship for **Steel A, Smooth Radii.**

Simulated gear tooth tests have been performed on steels A, B, and C of Table I. Four different T-shaped test blocks were made of steel A. Two of the blocks had 0.040-in. radius fillets and the other two blocks had 0.125-in. radius fillets. One block of each radius had smooth-finished fillets, whereas the fillets of the remaining two were purposely roughened with longitudinal tool marks. Stress-cycle relationships for this steel are shown in Fig. 10, and the endurance limits are tabulated in Table V. Figure 11 is a typical specimen failure. The failures originate in the fillets and appear to be identical to fatigue failures encountered in the roots of actual gear teeth.

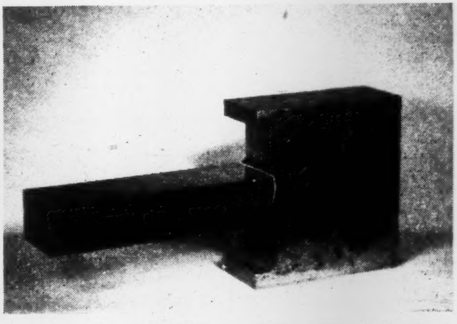


FIG 11.—Typical Simulated Gear Tooth Fatigue Specimen Failure.

Steel A was also tested in the case-hardened condition. The carburizing treatment consisted of pack carburizing a 0.125-in. smooth radius test block at 1650 F for 4 hr and furnace cooling to room temperature. After removal and grinding, the individual test specimens were heated to 1500 F, water quenched, and tempered at 200 F. Using this procedure, only the periphery was of high hardness. The approximate hardness and depth of case were 63 Rockwell C and 0.062 in., respectively.

As in the case of the contact roller tests, steel B was thoroughly investigated. Tests were made on this material in the following conditions:

1. As-received with 0.125-in. smooth fillets.

TABLE V.—SUMMARY OF SIMULATED GEAR TOOTH TEST DATA.

Steel	Type	Condition of Material	Hardness Brinell Hardness Number	Finish of Fillets	Simulated Gear Tooth Endurance Limit, psi		Rating Based on 0.125 in. Smooth Radius Fillet
					0.04 in. Fillet Radius	0.125 in. Fillet Radius	
A	SAE-1035	As-received	156	Smooth	20 000	31 000	1.0
A	SAE-1035	As-received	156	Rough	20 000	29 000	0.9
A	SAE-1035	Case hardened	Surface 710	Smooth	...	50 000	1.6
B	Ni-Mo	As-received	213	Smooth	...	35 000	1.1
B	Ni-Mo	As-received + shot peened	...	Smooth prior to shot peening	...	42 000	1.4
B	Ni-Mo	Induction hardened	Surface 415	Smooth	...	36 000	1.2
B	Ni-Mo	Induction hardened + shot peened	...	Smooth prior to shot peening	...	45 000	1.5
B	Ni-Mo	Flame hardened	Surface 480	Smooth	...	49 000	1.6
B	Ni-Mo	Flame hardened + shot peened	...	Smooth prior to shot peening	...	60 000	1.9
C	Cr-Ni-Mo-V	As-received	283	Smooth	...	39 000	1.3
C	Cr-Ni-Mo-V	As-received	283	Rough	...	35 000	1.1
C	Cr-Ni-Mo-V	Nitrided	Surface 560	Smooth prior to nitriding	...	67 000	2.2

2. As in 1 above, plus shot peening on the simulated tooth surfaces as follows:

Shot size	P-33
Shot type	chilled iron
Intensity	0.020 to 0.022 A2
Shot flow ratio	9 lb per min
Exposure time	½ min per in per fillet

3. Induction hardened with 0.125-in. smooth fillets.

4. As in 3, plus shot peening as in 2.

5. Flame hardened with 0.125-in. smooth fillets.

6. As in 5, plus shot peening as in 2.

The endurance limits for these conditions are shown in Table V.

Simulated gear tooth tests were made on steel C in the as-received condition with 0.125-in. smooth and rough fillets and in the nitrided condition with 0.125-in. smooth fillets. Nitriding was done at 975 F for a period of 100 hr. The pertinent data for these conditions and treatment are shown also in Table V.

Upon examining the results of the simulated gear tooth fatigue tests, one finds that the smaller fillet radius gave appreciably lower endurance limits. This was as expected. It is of interest to note, however, that in the case of steel A the intentional roughening of the fillets did not cause a marked lowering of the endurance limit below those of the smooth specimens. These results may be attributed to the high ductility and low hardness of steel A. As such, any detrimental stress concentrations produced by poor finish or intentional notches are relieved by the plastic flow of the material. The ability for this self-stress relief decreases with decreasing ductility and increasing hardness:

In general, flame hardening or induction hardening of the fillet areas have been applied only to comparatively small, coarse pitch gears. In fact, considerable difficulty was experienced in having the induction-hardened test specimens prepared. A simulated gear-tooth T-shaped test block was submitted for induction hardening. It was found necessary to cut the block into individual specimens before hardening because of the large amount of electrical energy required. Extreme caution should be used in interpreting the effectiveness of the flame and induction hardening treatments. The results reported herein can be interpreted only in terms of similar materials treated under identical conditions. As evidenced in Table V, flame hardening was effective in increasing the fatigue strength of the fillet, whereas induction hardening was not.

Whether or not flame hardening or induction hardening are effective is dependent upon the nature of the resultant surface stresses. These stresses may range from tension to compression and are determined by many factors such as material, mass, depth of heating, thermal gradient, hardenability, quenching medium, etc. Even where it can be applied, each particular application involving these treatments is a problem of its own and requires considerable investigation to develop the proper hardening procedure.

In direct comparison, case hardening and nitriding treatments generally introduce large compressive stresses in the surface which materially increase the fatigue strength. As shown in Table V, case-hardening steel A increased the fatigue strength by approximately 70 per cent. Likewise, nitriding steel C increased its fatigue strength by the same percentage.

Examination of Table V shows that shot peening is effective in increasing the fatigue strength of the root areas by approximately 25 per cent. An analysis of the stresses introduced by peening indicates that this treatment should be even more effective under conditions of unidirectional stress. Accordingly, an evaluation of some of the materials and treatments exposed to cyclic uni-directional stresses is currently under way, but the data obtained to date are too few to warrant reporting at the present time. A Krouse direct stress fatigue testing machine is being used for the uni-directional tests.

SUMMARY AND CONCLUSIONS

The first phase of the investigation of marine propulsion gears, dealing with the pitting resistance of ferrous, non-ferrous, and non-metallic materials, has yielded considerable information as to the expected behavior of various materials and treatments under rolling contact. The second phase has yielded information as to the effect of various ferrous materials and treatments on the fatigue strength of the teeth in the root areas.

The contact roller tests described in this paper have produced pits which appear to be identical, except for size, with those found in actual gear failures. For ferrous and non-ferrous materials, a stress-cycle relationship can be established and a so-called "pitting limit" determined for the various materials and treatments. In general, stress-cycle curves for ferrous materials exhibit a "knee," whereas non-ferrous materials do not. The pitting limit increases linearly with increase in hardness for through-hardened materials. The pitting limits for case hardening treatments such as carburizing and nitriding are the highest of any materials tested. These results are higher than would be expected from the pitting limit *versus* hardness relationship. This is undoubtedly due to the high compressive stresses initially present in such hardened cases. Chromium plating is not considered satisfactory as a hard surfacing material for highly stressed gears because of its tendency to spall under contact loading.

A limited number of tests indicate that the contact roller test is a suitable procedure for differentiating between various surface finishes. It appears that the pitting of a gear material is influenced to a great extent by the initial surface roughness of the material, and for any one ferrous material, the smoothest surface will have the highest pitting resistance.

Hardened copper-beryllium alloy has the highest resistance to pitting of any of the non-ferrous materials tested. The wrought aluminum alloy is superior in pitting resistance to the cast aluminum alloys and the Mg-Al-Zn alloy is superior to the Mg-Ce alloy. Aluminum, magnesium, and copper-base alloy gears must be mated with ferrous or non-ferrous gears of high hardness to prevent scoring of the tooth surfaces.

In testing nonmetallic materials, it was necessary to alter the criteria for failure because of the heterogeneity of the plastic-base materials. Instead of a pitting limit, a so-called "breakdown limit" was determined for these materials. It was concluded that the filler laminae of the plastic base materials must be oriented in planes perpendicular to the contact surfaces and that materials constructed of finely woven fabric have higher load-carrying capacities than those of coarsely woven fabrics.

The simulated gear tooth tests described in this paper for ferrous materials have shown that increasing the fillet radius will result in an increase in fatigue resistance of the gear tooth. Therefore, the root radius should be as large as is compatible with the tooth design. The application of shot peening increases the fatigue resistance of the root area by some 25 per cent. Induction hardening showed no improvement in root strength whereas flame hardening did. The results obtained by either process are greatly influenced by material, size, hardening procedure, etc. The most effective tooth strengthening treatments are carburizing and nitriding. When properly applied, both treatments increase the root strength by some 70 per cent.

It is important to recognize that the information contained in this paper permits only a qualitative comparison of the pitting resistance and root fatigue strength of the various materials. The numerical figures have little significance in actual gear calculations since their values are dependent upon the geometry of the specimen and the criteria used in designating a failure. The value of the data lies in providing an estimate of the expected behavior of various materials and treatments based on the performance of gear materials which have been used in large gears for many years. With but few exceptions, the conclusions reached herein correlate favorably with the as yet incomplete large scale destructive tests being conducted elsewhere and mentioned previously. Therefore, it is believed that the test procedures described and the ratings shown in Tables I and V will serve two purposes. First, they will be useful in designing future higher speed, lighter-weight gears for marine propulsion and other purposes; and, second, they will be useful in screening new materials and treatments as they develop or are required.

DISCUSSION

MR. R. E. Peterson.[1]—It may be of interest to mention a point or two regarding the mechanism of pitting. Using a roller test similar to that of the author, we found it impossible to produce pitting when the rollers were run with the rolling surfaces dry. We applied very heavy loads to these rollers, sufficient to cause the rollers to flow plastically, but no pits were produced. Then Mr. A. H. Burr found by accident that a lubricant would produce quite a striking effect. There was a leak in the apparatus so that some oil got down on the rolling surfaces, and then it was found that pitting did occur and that the load could be reduced to a fraction of what we had been trying to use, about a fourth, and still produce pitting with no plastic flow whatsoever.

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A hydraulic wedge theory was developed by Stewart Way. Small cracks making an angle with the surface develop, oil enters the cracks, and if the direction of rotation is such as to close the crack mouth as it enters the contact zone, a high hydraulic pressure develops within the crack as it passes through the contact zone, causing the crack to spread into a "conchoidal pit." Way even checked this by drilling a small hole in back of the cracked portion, and since this relieved the pressure, the crack growth ceased. Also, he changed the direction of rotation and caused the cracks to grow with opposite angularity. These general features of "pit" development seem fairly well established.

One or two other points: It was found that, in general, the pitting limit is increased with the hardness of the material; also, that the surface finish is important, the finer finishes giving higher pitting limits.

MR. J. O. ALMEN.[2]—The fatigue tests reported by Mr. Gross were intended to evaluate material for navy propulsion gears. Whatever value these tests may have, they certainly do not provide valid data by which material may be selected for use in gears. At best the data refer only to the particular cantilever specimens used in the tests; they are in no sense related to the probable behavior of navy gears made from the same materials. Short cuts based upon "simulated" specimens fatigue tested by "simulated" loading procedures as described by Mr. Gross can only give misleading data.

The fatigue strength or, more accurately, the fatigue durability of gears, whether for ship propulsion or for other uses, can only be measured by full scale tests of gear assemblies, including normal shafts, bearings, and gear cases. For accurate data the tests must be conducted by operating each assembly in actual service under normal operating loads. However, since nothing can be learned regarding the fatigue durability of any machine part unless the test terminates in failure, recourse must be had to less accurate laboratory tests in which overloads can be applied.

Accelerated laboratory tests, like service tests, must be made on full-scale normal assemblies because the fatigue strength of each gear in the assembly is influenced by the characteristics of each supporting member, including the gear case and its external supports. However, many errors are unavoidable in accelerated laboratory tests as compared to normal service operation.

Causing gears to fail by applying overloads introduces a number of errors that will lead to serious misinterpretations unless the nature of these errors is understood and their relative effects are properly evaluated.

1. Since all structural materials are elastic, any change in load involves changes in shape of all loaded members in the assembly. One result of these elastic deformations is that the tooth contact conditions, and therefore the concentration of loads, are not the same as those prevailing when normal service loads are applied. If the loads are uniformly distributed over the tooth width

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[2] Research Consultant, Research Laboratories Division, General Motors Corp., Detroit, Mich.

under normal loads they cannot be so distributed when overloads are applied. In fact, contact conditions can be correct for only one load and they will, therefore, be incorrect for any greater or lesser load.

2. For any concentration of load on a gear tooth the bending stress, which leads to tooth breakage, varies directly as the applied load, but the compressive stresses on the surfaces of the teeth, which lead to failure by pitting or scoring, vary as the square root or perhaps as the cube root of the applied load.

Other nonlinear stress changes that vary with the applied load develop through plastic movements within the loaded members. Plastic changes occur because the elastic limit of our structural metals is greatly reduced when subjected to repeated loads.

3. The bending fatigue strength of gear teeth may increase with use and with stress magnitude as a result of the development of favorable residual stresses. Gear teeth subjected to unidirectional loads may become permanently bent in the direction of the applied loads. Such deformation is rarely great enough to be noticed or to affect the functioning of a gear, but the resulting residual stress may greatly increase the fatigue strength of the teeth by reducing the effective tensile stress.

4. Gear teeth that will develop favorable residual stresses under unidirectional loads cannot acquire this advantage when subjected to reversed bending loads of equal magnitude. In fact, any favorable stresses initially present will tend to disappear under reversed loading, and fatigue strength will, therefore, also diminish with use. These effects are, of course, accentuated by overloads.

The practice of testing gear sets by closed circuit loading which in some cases applies reversed loads to gears that are normally loaded only in one direction can lead to wrong conclusions and bad designs.

Adequate fatigue data on gears must take into account the scatter that is inevitable in "identical" machine elements. Tests of full scale gear box assemblies by the Research Laboratories Division, General Motors Corp. on commercially indicated units show that this fatigue durability under identical loads varies more than ten to one when only about one dozen specimens are tested. For larger samples, the scatter becomes greater. Similar variations in fatigue durability must be expected of any machine part, including marine propulsion gears.

MR. M. R. GROSS (*author's closure*).—The author appreciates the comments of Messrs. Peterson and Almen. In regard to Mr. Almen's criticism of the investigation, there is no question that the "proof of the pudding" lies in a test of the full scale component as no simple laboratory test has or ever will be devised which will take into account the many parameters associated with as complex a structure as a reduction gear. As mentioned previously, tests have and are still being conducted on full size gears incorporating essentially the same materials and treatments described in the paper. It was the purpose of the investigation to perform simple laboratory tests in order to determine whether or not such tests could be used for evaluating and screening materials and treatments for large gears. Fortuitously or not, the laboratory tests have correlated in a relative way with the behavior of the large gears.

JOINT DISCUSSION OF PAPERS BY HAAKON STYRI AND M. R. GROSS

Mr. O. J. Horger.[1]—We have heard two papers this evening and there appears to be a great disparity between results and conclusions. First, Styri stated that his contact failures initiated below the surface. Gross offered no statement but Peterson in discussing the Gross paper explained that failure could start at the surface by means of a lubrication phenomenon presented by Way.[2] Therefore, two fundamentally different concepts of the mode of failure have been submitted.

Second, Styri shows *S-N* curves for contact stresses which do not flatten off at even 100 million cycles and exhibit considerable scatter. Gross on the other hand exhibits typical curves which flatten after 10 to 20 million cycles, and the scatter is comparatively nil.

Third, Almen discussed healing which may be associated with my next question. Styri used some noise level as a criterion of failure and found failure initiating from inclusions generally below the surface in a region of maximum shearing stress. Gross used a certain area of pit and number of pits as a criterion of failure. Neither of these authors have given any relationship of their laboratory criterion of failure with what constitutes pitting on service parts which just begins to classify them as unserviceable. The mere presence of pits which do not materially progress does not interfere with service operation. We have found pitting of this type and yet these parts were continued in service or in laboratory tests to many times the life at which this pitting would have classified the parts as failures by the criterion of the authors.

Chairman T. J. Dolan.[3]—I do not believe that we can settle now this particular problem that Mr. Horger has raised. It certainly is an interesting controversy, but I do not believe that we are quite as far apart in our answers as might be assumed. For example, I think that it might well be that many of these failures are initiating as subsurface failures at very small distances below the surface, which, in progressing to the surface, could then become visible by means of the mechanism which was described by Mr. Peterson. Certainly a crack initiating a few thousandths of an inch below the surface and progressing to the surface is difficult to detect and difficult to attribute either to a surface or a subsurface failure until it may subsequently be opened to a larger extent.

Also, in connection with this question of whether an endurance limit exists or not, I believe in Mr. Styri's paper he approached something that looked like an endurance limit for the softer metals, but it was only for these high-hardness ball bearing steels that he had no indications of actual leveling off of the data. I have not examined Mr. Gross' data in great detail but it is my impression that most of his results were on the softer metals which are more likely to exhibit definite endurance limits.

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[1] Chief Engineer, Railway Division, The Timken Roller Bearing Co., Canton, Ohio.

[2] S. Way, "Pitting Due to Rolling Contact," *Journal Applied Mechanics*, June, 1935, pp. A49–A58.

[3] Research Professor of Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

MR. HAAKON STYRI (*author's closure*).—In reference to Mr. Horger's comments on the two papers, I think that there is too great a difference in material tested and in test conditions to be able to make any valid comparison. We tested ball bearings with hardness over 60 Rockwell C, and at contact loads much higher than the highest used by Mr. Gross, even for his hardest material. The contact points in our tests were either circular, as in the No. 1207 outer races, or elliptical, as in No. 6207 inner races, with the maximum contact stress at the center. The contacts in Gross's tests were rectangular. Failures in the ball bearing races will develop rapidly from an initial pit, and simultaneous failures at more points are relatively few. We have run tests on deep groove inner races made of soft aluminum and found many starting points for spalling. In roller bearings, stresses at one initial failure may be relieved, and a major failure develop at another point. Fatigue failures in laboratory tests of bearings have the same characteristics as fatigue failures in the field, and operating conditions will tell when they should be taken out.

It seems difficult to determine scatter and fatigue limits from the few tests run by Gross if, as indicated in his Fig. 5, only one test is made at each load and the test at the lowest load is stopped at about 11,000 cycles. In the hard specimens no failures at all are reported.

The tests which we have reported were run with the purpose of finding some simple test that would throw light on the causes of fatigue failure in ball bearings.

It has been shown clearly that damage starts below the contact point in ball bearings, and very often at inclusions of the oxidic type. But inclusions are not the only cause for start of spalling, and they are not the only reason for high scatter in fatigue life in hardened steel test specimens under high stresses, as shown in tests run on specimens and bearing parts made of vacuum melted steel.

In our tests on ball bearings or on simple fatigue specimens, no fatigue limit has yet been found even with runs beyond 10^9 cycles to failure, but this does not prove that there is no such limit. For lower hardness, as Mr. Dolan says, both in rotating-beam and torsion-type specimens, there seem to be well-established fatigue limits, depending on the heat treatment, and we should certainly expect high scatter of lives in the neighborhood of the fatigue limits as long as we do not operate with absolutely homogeneous and stress-free material.

Just as Gross indicates an increase in strength of specimen, when reversed bending has not caused failure beyond 10^7 cycles (his Fig. 10), we also found such indication in the torsion test of the softer grades around 30 Rockwell C (my Fig. 9).

When Mr. Way's[2] paper was published in 1935, extensive comments were made by me showing that spalling could well occur when lubricant was not present and that we often found cracks that start below the undamaged surface, but near the loaded area.

THE FATIGUE TEST AS APPLIED TO LEAD CABLE SHEATH*

By G. R. Gohn[1] and W. C. Ellis[1]

Synopsis

This paper discusses the more important factors affecting the design of laboratory test methods suitable for obtaining significant fatigue data from reversed bending tests on cantilever-beam specimens of lead cable sheathing alloys. Data are presented to show the effect of cycling rate, temperature, shape of specimen, alloy additions, and aging on fatigue life. The close correlation between bending fatigue tests on strip specimens and full size sections of cable is demonstrated. The fatigue data are analyzed in terms of (*1*) cycle life *versus* deflection, (*2*) cycle life *versus* strain, and (*3*) cycle life *versus* stress. Photomicrographs illustrating representative laboratory and field failures are included in the paper.

The association of cracking in lead cable sheath with cyclic strains in the field was clearly recognized in a paper by J. R. Townsend (**1**)[2] in 1927. He described fatigue machines with which similar cracking was obtained in the laboratory and demonstrated the superiority of alloyed lead in resisting cracking. This pioneering paper was followed later in 1927 and in 1928 by four papers (**2, 3, 4, 5**) in Europe setting forth the European experience with very much the same conclusions. Since then there have been many studies on the nature and cause of fatigue failure in lead alloy sheath until today there is an extensive literature (**6**) on fatigue as well as on the other properties of lead alloys determining their suitability for sheathing applications.

For a well-rounded appraisal of the suitability of an alloy for sheathing purposes, a knowledge of many physical properties is required. Among these properties are: plasticity at extrusion temperatures, strength, resistance to abrasion and corrosion, aging characteristics, creep strength under steady stress conditions, fracture characteristics when subjected to stressing at low strain rates, and resistance to fatigue failure. While all of these properties are important, the present paper is limited to a discussion of methods for determining fatigue properties and to a presentation of test results for some representative lead alloys.

Origin of Fatigue Failures in Cable Sheath:

Fatigue failures in telephone cables are confined almost entirely to aerial installations in which the cable is attached to steel suspension strand supported by poles. These failures are caused by daily and seasonal changes in temperature that set up cyclic strain in the sheath, particularly in bows near the poles (**1**). Much effective work has

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[*] Presented at the Fifty-fourth Annual Meeting of the Society, June 18–22, 1951.

[1] Member of Technical Staff, Bell Telephone Laboratories, Inc., New York, N. Y.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 739.

been done on suspension methods designed to reduce the magnitude of these strains. Since telephone cables, buried or installed in ducts, are essentially at constant temperature, variations in strain due to thermal changes are generally negligible. In the case of power cables (most of which are in ducts) the situation is different: here the cyclic strains result from longitudinal movement of the cable caused by temperature changes arising for the most part from variations in the power load (7). The cycles of strain induced in the sheath depend upon local conditions. However, in both telephone and power cable the time for a normal strain cycle is large compared to that present in the usual machine designs where fatigue is a determining factor; for example, a rotating shaft or a flexing spring.

Another type of cyclic strain sometimes encountered in service is of a higher frequency but of a lower amplitude than that due to thermal changes (1, 2). This results from vibration and occurs either during transportation of the reeled cables or, in some instances, on cables strung on bridges or structures subjected to heavy traffic.

Choice of Test Conditions:

The design of a fatigue test for lead is influenced by some factors peculiar to its properties and the unique service conditions of the cable. First, lead alloys melt at low temperatures, and hence fatigue tests on lead alloys are relatively high-temperature tests. At such temperatures, lead alloys are strained in the plastic range, even at the very low strains imposed either in service or in the laboratory. Under these conditions, stress can be calculated from the bending moment for a given deflection, but such stress values are time-dependent. This time-dependence of stress points to one of the advantages of analyzing fatigue data in terms of strain range rather than stress.

There is another reason for correlating the fatigue life of cable sheathing alloys with strain range; this relates directly to service conditions. As mentioned previously, environment or service loads impose the largest cyclic strains in sheath. The magnitude of these strains is not limited by the inherent strength of the lead, hence stress correlations are secondary to strain correlations.

Since the strain range extends into the plastic region and, since lead alloys are used at temperatures where creep (slow deformation) is significant, the life of the fatigue specimen will frequently depend on the rate of cycling. This has been recognized and studied by many investigators (8, 9, 10, 11). These studies, as have our own, indicate that the cycle rate for most significant information should be chosen to give results similar to those found in the field. If temperature changes produce only one cycle of strain per day in the field (as in the case of telephone cables), then the preferred test rate is one cycle per day. Since so slow a test is not practicable, we have compromised on 15 cycles per hour.[3] With this acceleration, the types of fatigue fractures are significantly the same as those found in the field.

So far as we know, no attention has been paid to the effect of different strain-wave forms or rest periods in the fatigue testing of lead. We have used a simple sinusoidal shape which may be defined by the range of the maximum strain (twice the strain amplitude) and the frequency. Such a sinusoidal shape is, at best, only an approximation to the cycle shape induced by the daily temperature variation in the field. Here the strain usually changes rapidly in a short period followed by a longer period of practically no change. Studies of the effect of strain-wave shape and rest periods are needed.

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[3] This is equivalent to ¼ cycle per minute (cpm) or 360 cycles per day.

The choice of ranges of strain is again dictated by service conditions and the time available for conducting the test. However, the test used should not depart significantly from the conditions of service. This requires that the ranges studied include those of the field conditions and implies a knowledge of the strains occurring in the field under different methods of installation and for various daily and seasonal changes.

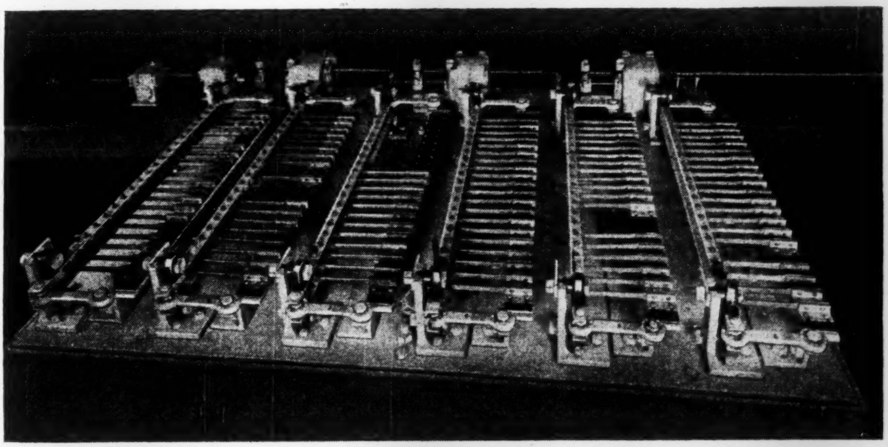

FIG. 1.—126-Specimen Cantilever-Beam Fatigue Machine for Testing Cable-Sheath Alloys at Six Different Deflections.

The fatigue life of lead and lead alloys depends on the temperature of test, as will be shown later. This raises the question of the preferred testing temperature if the tests are to be made at only one temperature. We have used 80 to 85 F as the test temperature, a temperature higher but not seriously out of line with average service conditions (12). Of even more importance than the actual test temperature is the maintenance of a reasonably constant temperature throughout a series of tests. Only by this refinement can reliable comparisons of alloys be made.

Finally, the type of test sample must be selected: will it be a length of cable or a specimen cut from flattened sheath or rolled sheet? Both types of specimens yield useful information and are frequently used to supplement each other (as we do in our own laboratory and as has been done by Moore and others (6, 8, 9, 13)). The strip specimen is particularly useful in tests for sorting alloys, in developing new compositions, and in making an evaluation comparing sheathing alloys. Tests on sections of full-size cables can be made only after such cables are manufactured. Since service conditions can be closely simulated, results from such tests are, in general, preferred by engineers engaged in predicting service life.

The type of test that uses a section of cable is required for appraising composite sheaths made from plastics and lead alloys or for evaluating the effect of core structure on sheath life. Individual tests on components of composite sheath are of only limited value, because the constraints and the complex loading imposed on individual components usually cannot be analyzed satisfactorily.

The preferred methods for the fatigue testing of lead described in this paper have been arrived at after 25 yr of experience in comparing the results obtained in the laboratory with field service. In developing these methods, the most important consideration was the simulation of field conditions as closely as possible consistent with a useful output of information.

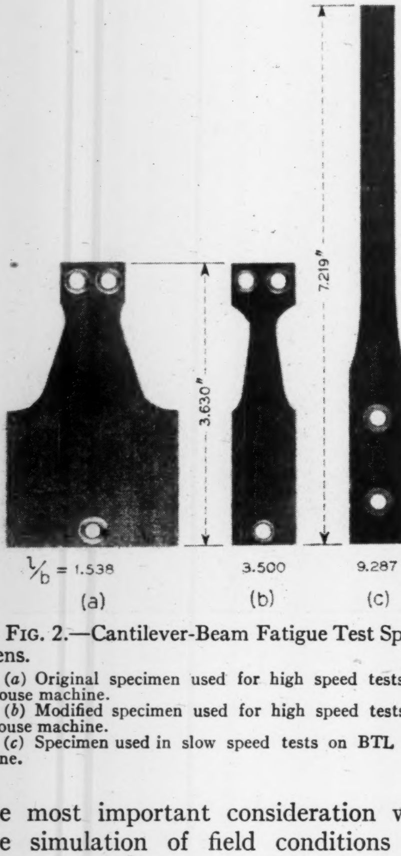

FIG. 2.—Cantilever-Beam Fatigue Test Specimens.

(*a*) Original specimen used for high speed tests on Krouse machine.

(*b*) Modified specimen used for high speed tests on Krouse machine.

(*c*) Specimen used in slow speed tests on BTL machine.

METHODS OF TEST

Fatigue Machines:

Two general types of fatigue testing machines are used: (*1*) those for flat cantilever-beam specimens and (*2*) those for full-size cable sections. The fatigue machine used for testing flat cantilever-beam specimens at slow operating speeds ($\frac{1}{4}$ cpm) is shown in Fig. 1. This machine is the culmination of a series of machines designed by Townsend (**1, 14**) and Greenall (**12, 14**). In this machine the specimens, Fig. 2(*c*) are mounted horizontally. Modifications in which the specimens are mounted vertically are also used. The latter have been enclosed in boxes and are operated at controlled temperatures differing from 80 to 85 F for high- and low-temperature tests. Various deflections or strain ranges may be obtained by changing an adjustable cam controlling the stroke of the reciprocating arms. Likewise, the machines have been operated at rates of cycling varying from one cycle per day to 2000 cycles per minute; at the present time all tests on these machines are made at $\frac{1}{4}$ cpm (360 cycles per day).

For tests at high rates of cycling (1650 cycles per minute), a modified Krouse flat-plate single-specimen machine is used. The modifications consist essentially of a stationary specimen-clamping block permitting the use of shorter fatigue specimens such as those shown in Figs. 2(*a*) and (*b*); a counterbalanced magnesium alloy clamp at the moving end of the test specimen to reduce inertia effects and to insure simple bending; and a safety bumper to prevent damage to the broken ends of the specimen after failure. These modifications permit testing strip specimens of lead alloys having a thickness of 0.050 to 0.140 in.

All of the fatigue tests are made in reversed bending (zero mean strain). The end point is either fracture or 100 million cycles of strain without failure.

For sections of cable, two machines are used.[4] One of these, the cable-bending machine, shown in Fig. 3, was developed by G. M. Bouton for the reversed bend testing of full-size sections of cable (**17**). In this machine a 6-ft length of cable, either with or without the core, is loaded at the quarter points by means of two movable arms. These arms produce a constant but adjustable strain in the section of cable lying between them; the cable is held in this strained position for a predetermined period of time, after which the deflecting force is removed, the cable rotated 180 deg, and the deflecting force again applied by motor-driven cams. This causes a reversed bending of the cable through a fixed cycle of operations. By adjusting the throw of the actuating load arms, various strain ranges can be obtained within the test length. This machine, as G. M. Bouton has demonstrated,[5] in the course of the fatigue test produces buckles in the sheath, a condition frequently observed in the field.

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[4] Other machines for testing full-size sections of cable have been described by Townsend (**1**), Greenall (**12**), Roper (**15**), Halperin (**16**), Moore and Dollins (**13**), Chaston (**18**) and others.

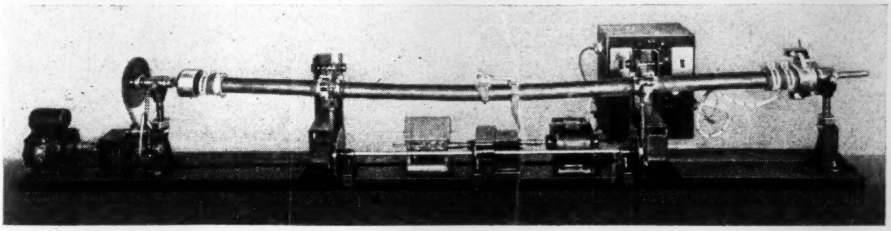

FIG. 3.—Cable Bending Machine for Fatigue Tests on Full-Size Sections of Cable (with mechanical extensometer in place for strain measurements).

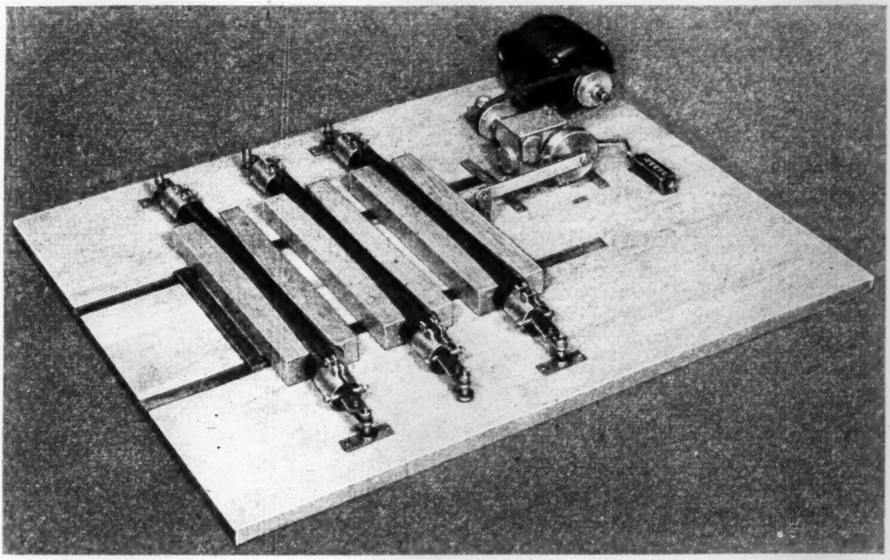

FIG. 4.—Constrained Arc Machine for Fatigue Tests on Full-Size Sections of Cable.

Another type of machine (**17**), the constrained arc machine, developed by J. D. Cummings for testing full-size sections of cable, is that shown in Fig. 4 Here the cable is forced into the predetermined curvature of a pair of wooden blocks by the reciprocating motion of the blocks in a horizontal plane. Blocks of different curvatures are used to obtain a series of strain ranges. This type of machine is useful for testing both lead sheath and composite types with plastic outer jackets.

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[5] Private communication.

Stress Determination:

To determine the correlation between stress and cycle life, reversed-bending tests are made at various amplitudes on cantilever-beam specimens like that in Fig. 2(*c*). While the specimens are cycled at a constant rate, the instantaneous bending moment is measured at the point of maximum deflection. From this instantaneous bending moment and the known constants of the cantilever-beam specimen, the maximum bending stress can be calculated. Data for these calculations may be obtained from a machine such as that described by Boettler and Werring[6] or from modified commercially available stiffness testers.

Strain Measurements:

Strain ranges for the strip specimens are measured with SR-4, Type AD-7, strain gages cemented to the critical section of three representative samples of each alloy. All gages are attached with nitro-cellulose cement.

The strain ranges are determined by deflecting the free end of the test specimens through the chosen amplitude. The procedure used is to determine first the strain range for the minimum amplitude and then to repeat the determination for each successively greater amplitude. The effect of the previous deflection at a smaller amplitude is negligible because, after making strain readings at the higher amplitudes, it was possible to return to the lower amplitudes and check the initially recorded strain readings. Furthermore, at low and intermediate amplitudes the strain readings are constant through periods of 4 hr or more for repeated readings made both at $\frac{1}{4}$ and at 1 cpm.

The same method is used for evaluating the total strain per cycle for the specimens tested at 1650 cpm except that the specimens are deflected in the Krouse machine by hand operation. This technique was only adopted after experiments in which dynamic strains were measured had shown that the total strain per cycle was substantially the same for specimens tested at 1650 cpm as for specimens tested at $\frac{1}{4}$ cpm.

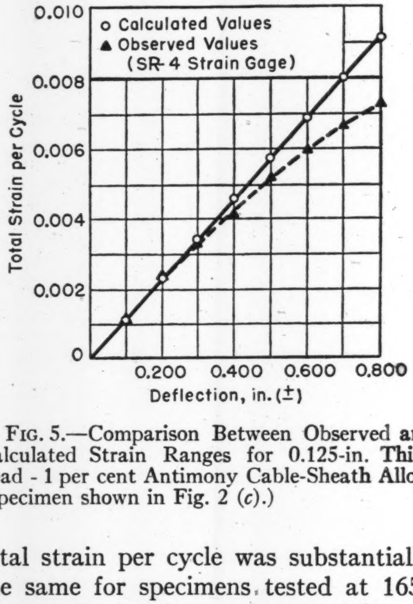

Fig. 5.—Comparison Between Observed and Calculated Strain Ranges for 0.125-in. Thick Lead - 1 per cent Antimony Cable-Sheath Alloy. (Specimen shown in Fig. 2 (*c*).)

There are some disadvantages to using wire gages for these strain measurements. These arise principally from a small but significant stiffening of the section to which the gage is cemented. Reliable fatigue data cannot be obtained with specimens to which strain gages are attached, because the specimen invariably fails at the end of the paper of the gage rather than within the critical section. This effect is reduced by closely trimming the paper around the gage and by the use of a minimum amount of cement. The stiffening effect[7] introduces an error in the strain determination. This is less on wide specimens such as those shown in Fig. 2(*a*), than for those shown in Fig. 2(*c*), but no significant difference was found between specimens shown in Fig. 2(*a*) and (*b*). However, some quantitative studies of the magnitude of this error made on cantilever-beam specimens having a uniform width of 0.403 in. and a thickness of 0.125 in. indicate little difference between the calculated and observed strain for ranges up to 0.5 per cent. The magnitude of the error is shown in Fig. 5.[8] As may be seen from these two curves, in the strain ranges of greatest interest, namely, 0.005 strain and below, the agreement between the

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[6] U. S. Patent 2258276, October 7, 1941.

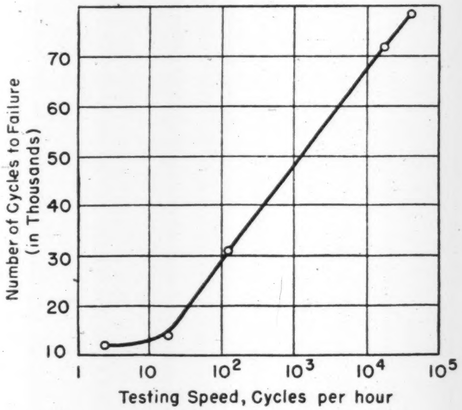

FIG. 6.—Effect of Testing Speed on Cycle Life of Lead - 1 per cent Antimony Cable-Sheath Alloy.

Tested in reversed bending at 85 F, zero mean strain and 0.004 total strain.

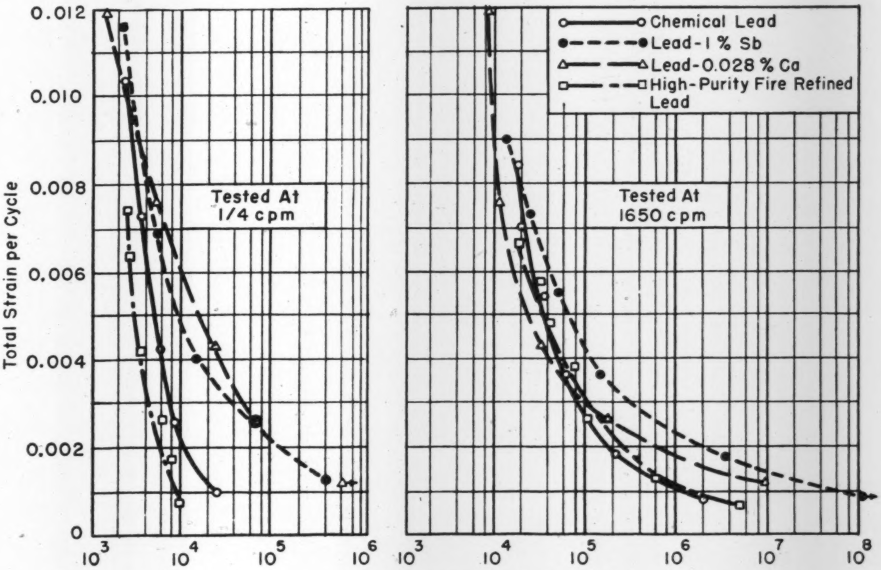

FIG. 7.—Effect of Testing Speed on Cycle Life of Various Cable-Sheath Alloys.

Tested in reversed bending at 85 F.

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[7] There may be an additional effect resulting from the cementing of the gage to the specimen. The addition of plastics to the surfaces of lead fatigue specimens has been shown to alter the fatigue life (**10**).

[8] The calculated strain values shown in Fig. 5 were obtained from the equation:

$$\varepsilon = \frac{3t\Delta}{l^2}\left(1 - \frac{x}{l}\right)$$

where:

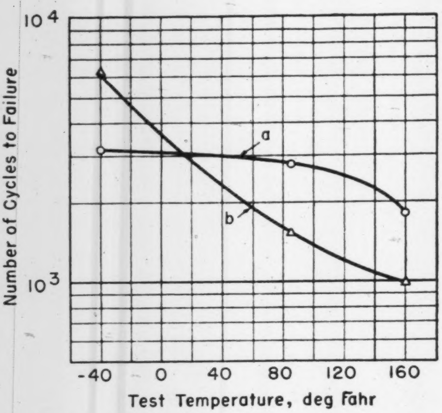
ε = strain for one-half cycle,
t = thickness in inches,
l = length in inches (distance from clamp to point at which deflecting force is applied),
x = distance from clamp to center of SR-4 gage, and
Δ = deflection at $x = l$.

theoretical and measured values is good. Above 0.005 strain the data on cycle life are conservative; that is, the cycle life is associated in the analysis with a strain less than that which is probably present in the specimens.

DATA AND DISCUSSION

Effect of Cycle Rate on Fatigue Life:

Laboratory tests were made to establish more precisely the relation between fatigue life (number of cycles to failure) and rate of strain cycling. Results of early studies by Babington on lead-1 per cent antimony cable sheath are summarized in Fig. 6. From these tests and others made at strain frequencies as low as one cycle per day, it was evident that the rate of cycling at frequencies greater than 10 to 15 per hour had a marked effect on lengthening cycle life. The cycling frequencies used by different investigators (**8**, **9**, **10**, **11**, **19**) have varied but most have used a low rate, particularly when simulating strain changes due to temperature.



(*a*) Lead-0.8 per cent antimony alloy cable sheath tested at 0.0106 strain.
(*b*) Lead-0.028 per cent calcium alloy cable sheath tested at 0.0116 strain.

FIG. 8.—Effect of Temperature on Cycle Life.

Tested in reversed bending at ¼ cpm and zero mean strain.

The effect of cycle rate on the fatigue characteristics of two lead alloys and two grades of lead for a series of strain ranges is shown in Fig. 7. An important finding was that the effect of cycle rate is more marked for some alloys than for others. Whereas the two grades of lead and the lead-1 per cent antimony alloy had about a ten-fold increase in fatigue life when the testing speed was increased from $\frac{1}{4}$ to 1650 cpm, the increase was less marked for the high strength lead-0.028 per cent calcium alloy. It is probably safe to generalize from this observation that the stiffer the alloy the less its fatigue life is affected by variations in cycle rate.

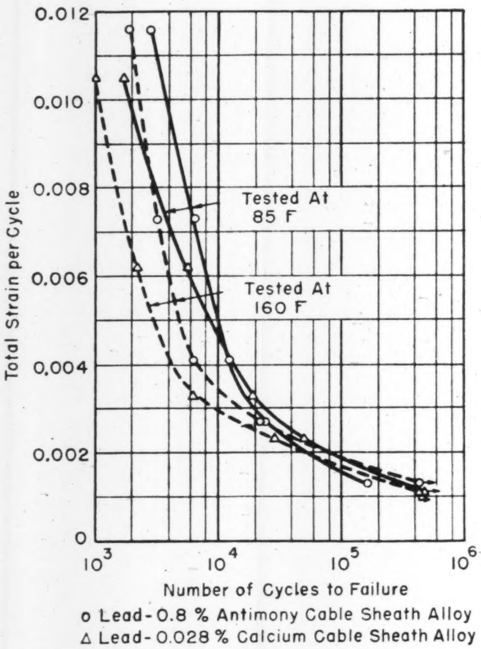


FIG. 9.—Effect of Temperature on Cycle Life.

Tested in reversed bending at ¼ cpm and zero mean strain.

Effect of Temperature on Fatigue Life:

The temperature of test has a marked effect on fatigue life but the effect again varies with composition. This is illustrated in Fig. 8 for two alloys tested at one strain range (a high range) over temperatures from −40 to 160 F. For the lead-0.8 per cent antimony alloy, increasing the temperature from −40 to 85 F resulted in a decrease in cycle life of only 14 per cent but a loss in cycle life of 33 per cent resulted when the test temperature was raised from 85 to 160 F. For the lead-0.028 per cent calcium alloy the loss of cycle life with temperature was continuous from −40 to 160 F. At this strain level (0.01) the calcium alloy had the greater life at the lowest temperature but this superiority was lost at 160 F.

The effect of temperature was investigated in greater detail for a series of strain ranges at temperatures above 85 F. The results are summarized in Fig. 9. At intermediate and high strain ranges the cycle life of both sheaths is less at 160 F than at 85 F. For low strain ranges the cycle life is affected much less by this change in temperature. For example, when the strain range was reduced to 0.0013, the cycle life of the antimony sheath was greater at 160 F than at 85 F. The temperature effect is important in the plastic part of the strain. However, when the plastic part is a small fraction of the whole—as at low strain ranges, one would expect the effect of a change in temperature on cycle life to be comparatively small. This was observed.

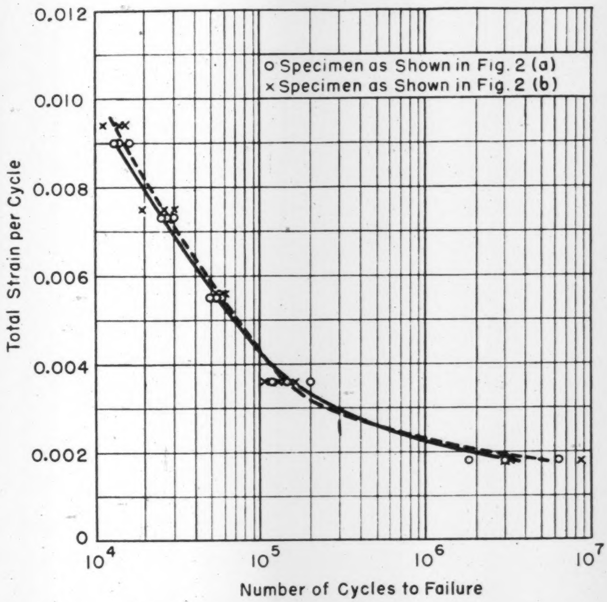


FIG. 10.—Effect of Shape of Specimen on Fatigue Life of Lead - 1 per cent Antimony Cable-Sheath Alloy.

Tested at 1650 cpm, 85 F and zero mean strain.

Shape of Test Specimen:

While the shape of the cantilever-beam fatigue test specimen undoubtedly affects cycle life, it is assumed that the effect is small; it may actually be of little significance. For example, most of the fatigue tests on the Krouse machines were made on specimens with the contour shown in Fig. 2(*a*). When it became necessary, in a sorting study on various alloys, to test specimens from

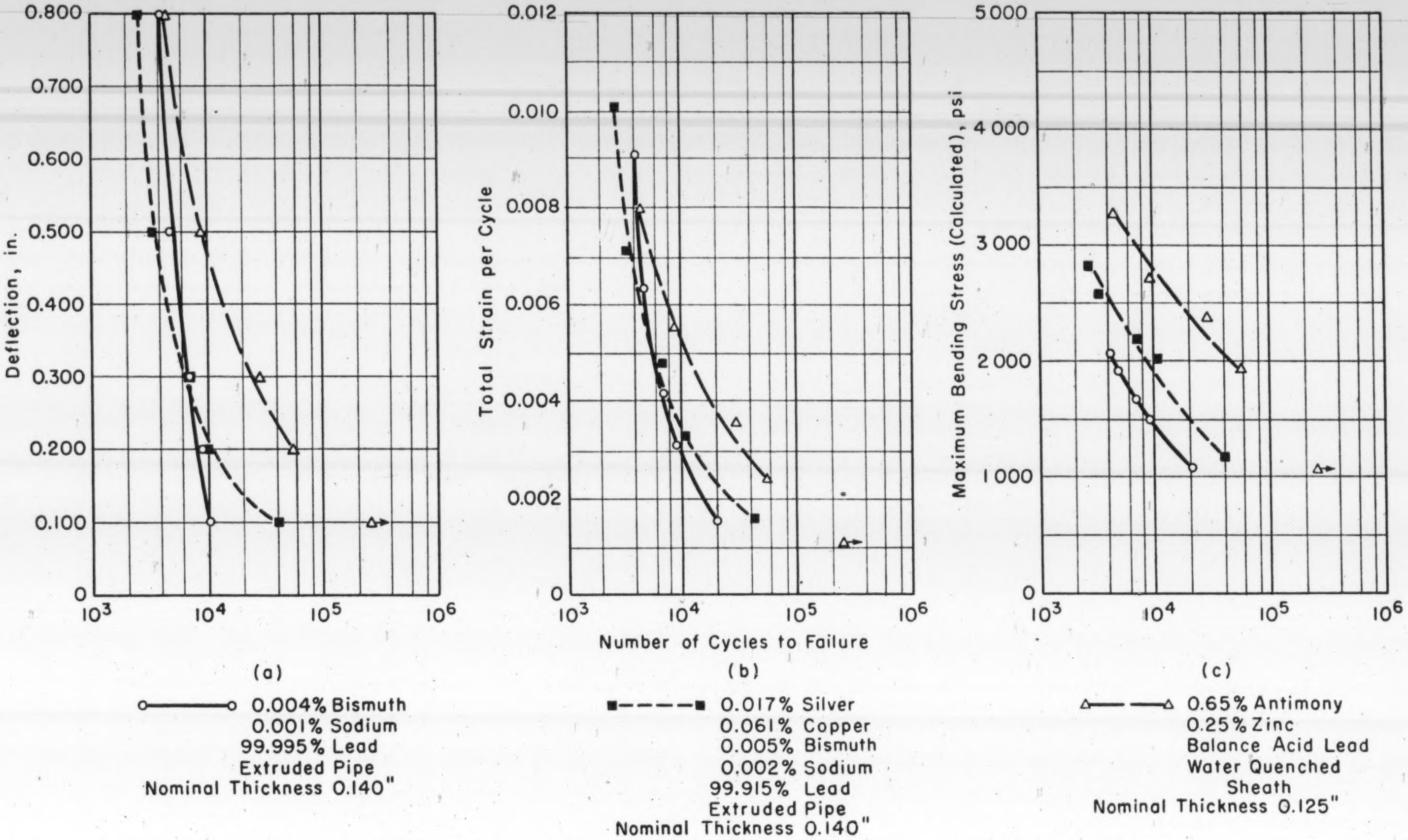


FIG. 11.—Different Methods of Analyzing Fatigue Test Data for Lead Cable-Sheath Alloys.

(*a*) Deflection basis.
(*b*) Strain basis.
(*c*) Stress basis.

Tested at ¼ cpm, 85 F, and zero mean strain.

$\frac{3}{4}$ in. wide extruded tape, the narrow specimen shown in Fig. 2(*b*) was developed. Tests on the same material but employing these two specimens show no significant difference in cycle life as may be seen in Fig. 10. Thus, for tapered cantilever-beam specimens of different shapes and having an l/b ratio within the limits of 1.5 to 3.5, reasonably close strain-cycle life relationships are obtained. Likewise, data for notched specimens of acid lead reported by J. F. Eckel (**11**) agree quite well with data given in Fig. 7 for the two unalloyed leads tested under similar conditions of strain and rate of cycling.

Analysis of Fatigue Data:

Fatigue data may be represented in three different ways: (*1*) cycle life for a series of deflections, (*2*) cycle life for a series of calculated maximum bending stresses, and (*3*) cycle life for a series of strain ranges. All have been used but, as discussed earlier, it is now generally considered that analysis on the basis of strain is most informative. The fatigue life for a high-purity lead, a low alloy lead containing small amounts of silver and copper, and a more highly alloyed lead-antimony-zinc alloy[9], all analyzed in three different ways are shown in Fig. 11. The latter alloy had a nominal thickness of only 0.125 in. as compared to a nominal thickness of 0.140 in. for the other two sheathing alloys. On a deflection basis (Fig. 11(*a*)), the lead-antimony-zinc alloy has a much superior cycle life due in part to the comparatively thinner specimen. The comparison on a deflection basis is misleading to a degree when specimens of different thickness are being compared. This limitation is removed when the data are presented on a strain basis, as in Fig. 11(*b*). The lead-antimony-zinc alloy is still superior in cycle life at low strain ranges but the superiority is reduced when the variation in thickness of specimen has been taken into account. On a stress basis (Fig. 11(*c*)) the lead-antimony-zinc alloy has superior cycle life at all stress levels. A stress representation usually unduly favors the stiffer, higher strength alloy as in this instance. Furthermore, the stress representation does not correspond to conditions imposed in the field, as discussed earlier, and is therefore of limited value in appraising alloys.

However, a stress representation is informative when the stress values are determined at successive stages in the life of the specimen. In this case details of the onset and progression of the fatigue failure are revealed. This has been done for a high-purity lead and the lead-antimony-zinc alloy. The data are plotted in Figs. 12 and 13. In some cases, particularly at high strain ranges, as in the case of the high-purity lead (Fig. 12), the stress increases at first with increased cycle life. This is probably associated with strain hardening. When the cycling is carried far enough, however, the stress falls off. This falling off in stress we attribute to fatigue damage. If it were due to recrystallization, one would expect a subsequent increase in stress—this was not observed. In other cases, as shown in both Figs. 12 and 13, any cycling at high stress ranges damages the material and causes a loss in resistance to the applied bending moment. Obviously, with variable stresses existing at different stages in the fatigue test, interpretation of fatigue data in terms of initial stress is of limited significance.

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[9] The lead-antimony-zinc alloy contained 0.65 per cent antimony and 0.25 per cent zinc. The specimens were taken from a commercial extrusion of sheath. This alloy was developed by G. M. Bouton and E. E. Schumacher.

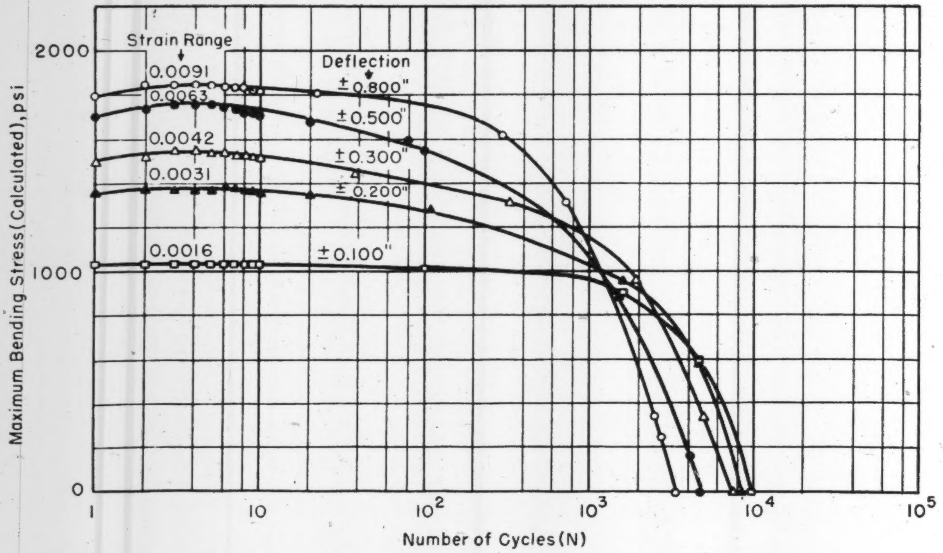

FIG. 12.—Cycle Life *versus* Calculated Maximum Bending Stress for High Purity Lead.
Extruded as pipe by Okonite Callendar Cable Co. Tested at ¼ cpm, 85 F, and zero mean stress.

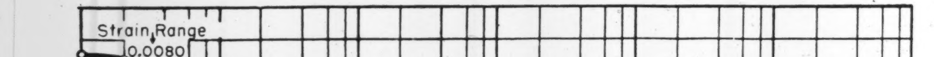

FIG. 13.—Cycle Life *versus* Calculated Maximum Bending Stress for Water-Quenched Lead 0.65 per cent Antimony - 0.25 per cent Zinc Cable-Sheath Alloy.
Tested at ¼ cpm, 85 F, and zero mean stress.

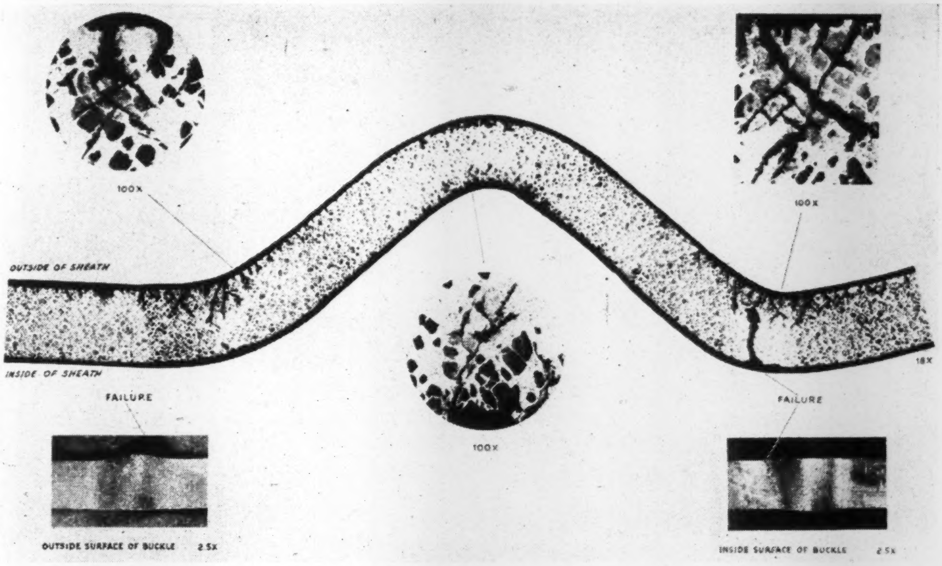

FIG. 14.—Sections Through a Buckle in a Field Fatigue Failure in Lead - 1 Per Cent Antimony Cable Sheath (Original magnifications as shown).

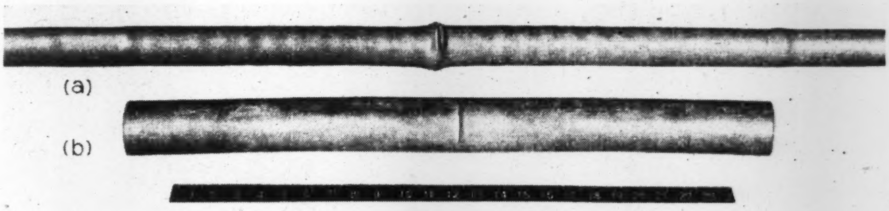

FIG. 15.—Sections of Failed Cable.
(*a*) Tested on cable bending machine (Fig. 3).
(*b*) Tested on constrained arc machine (Fig. 4).

Correlation Between Laboratory Fatigue Tests on Cantilever-Beam Strip Specimens and on Sections of Cable and Comparison of Laboratory Failures with Fatigue Failures in the Field:

Fatigue failures in the field frequently occur in aerial cable at poles where the sheath has buckled in the bows thrown up by seasonal temperature variations. Fatigue cracking in a buckle in sheath from an experimental pole line installation is shown in detail in Fig. 14. The center figure is a longitudinal section through the buckle depicting the areas of extensive fatigue cracking. These are regions of sharp radii of curvature and consequently maximum strain range when the cable lengthens and shortens. The cracking in relation to the grain structure can be seen in the several photomicrographs. The 45-deg cross-hatching of cracks, particularly evident in the photomicrograph on the right, is characteristic of fatigue failures in lead-antimony sheath. This pattern suggests that recrystallization occurs during the life of the cable in such a way that grain boundaries are aligned at 45 deg to the longitudinal strain prior to the formation of the fatigue crack. This, (as shown later) is also characteristic of the geometry of cracking in laboratory fatigue tests.

A completely satisfactory test on a full-size section of sheath should, in the course of cyclic straining, produce buckles just as temperature cycling does in the field. The cable-bending machine (Fig. 3) does this, as illustrated by the photograph of the longer sample in Fig. 15(*a*). There are numerous small buckles along the section of uniform strain and one large buckle in which a large fatigue crack appears. Bouton[10] has shown that the network of cracks in the buckle are similar to those in field failures. The shorter sheath sample in the same photograph (Fig. 15(*b*)) is a specimen tested in the machine shown in Fig. 4. There is a small buckle containing fatigue cracks near the middle of the test length. In these tests on full-size sheath, as soon as the buckle forms, the strain is intensified and the strain range is greater than that measured at the start of the test. Buckling does not occur in flexural tests on strip samples and therefore in this type of test (the cantilever-beam test) the strain remains sensibly the same through out the test period.

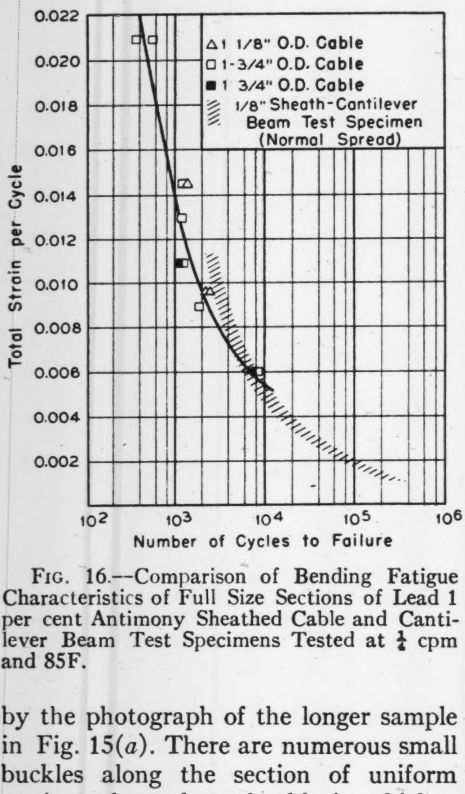

FIG. 16.—Comparison of Bending Fatigue Characteristics of Full Size Sections of Lead 1 per cent Antimony Sheathed Cable and Cantilever Beam Test Specimens Tested at $\frac{1}{4}$ cpm and 85F.

Despite the apparent difference in the progression to failure in the two types of tests, the strain range-cycle life relations are very nearly the same (see Fig. 16). In Fig. 16 the filled squares represent data for full-size sections of cable tested on the cable bending machine; the open squares represent data from full-size sections of cable tested on the constrained arc machine; and the shaded area represents the results of fatigue tests on cantilever-beam strip specimens made on the multi-specimen fatigue testing machine. At strain ranges from 0.006 to 0.012 the cantilever-beam test specimens have slightly longer lives than do the full-size sections of cable; at lower strain ranges the data from the strip specimens appear to be more conservative; that is, for a given strain range the cycle life is equal to or less in tests on strip specimens than in tests on sections of cable.

If the laboratory tests are to be significant, fatigue cracks in laboratory test specimens should appear the same as those observed in field failures. Some comparison of sections near the fracture are given in Fig. 17 for both slow and fast cycling rates on test specimens and from sheath failures in the field. The field failure for the slow cycle rate is the result of temperature changes; at the fast cycle rate from vibration during transportation. The appearance of the fatigue failure after a slow rate of cycling is nearly the same as that of the

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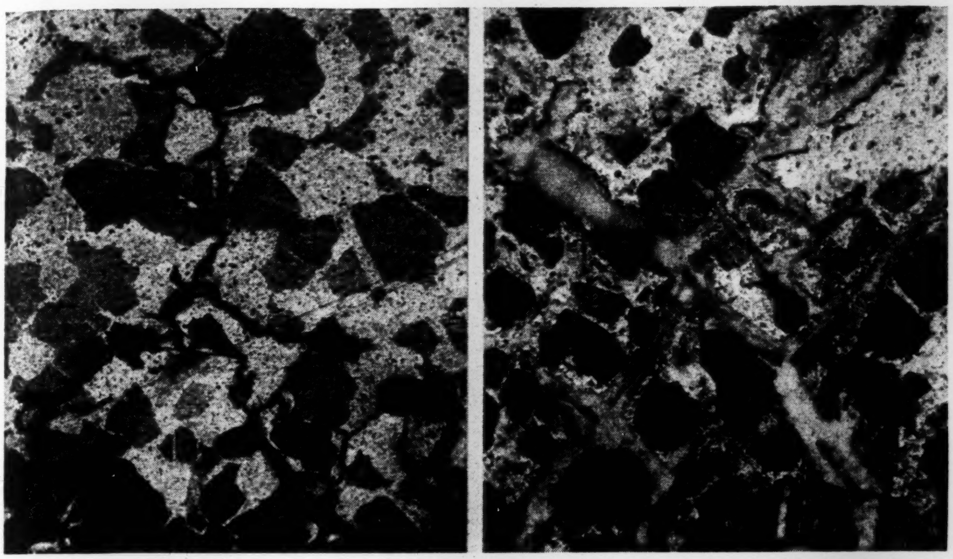
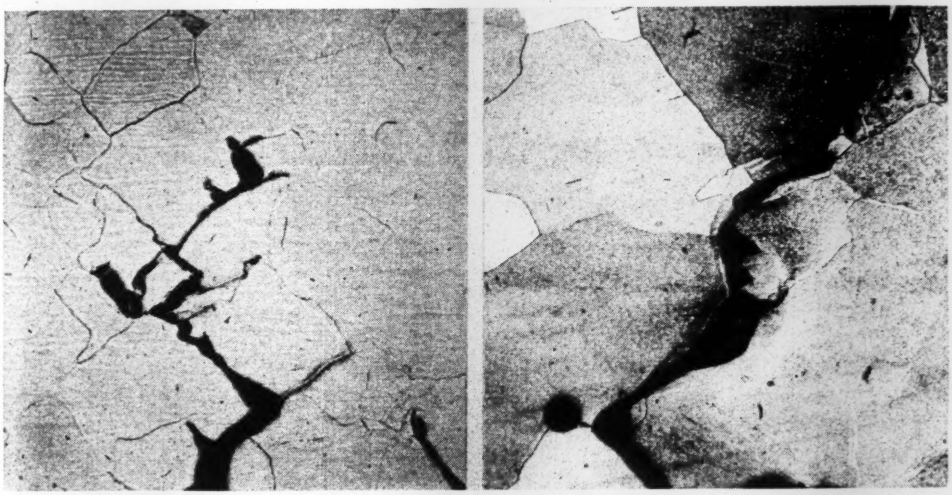
[10] Private communication.



(*a*) Lead - 1 per cent Antimony Alloy Sheath Tested in Laboratory at ¼ cpm, 0.0026 Strain, and 85 F. Failed After 51,300 Cycles. (Slow cycle-rate failure.)

(*b*) Lead - 1 per cent Antimony Alloy Sheath—Section Through Field Failure in Experimental Aerial Cable After 8 yr of Service. The Cracks Contain Corrosion Products. (Slow cycle-rate field failure.)



(*c*) Lead - 0.010 per cent Silver Alloy Sheath Tested in Laboratory at 1650 cpm, 0.0017 Strain, and 85 F. Failed After 114,000 Cycles. (High cycle-rate failure.)

(*d*) Lead - 0.010 per cent Silver Alloy Sheath—Section Through Failure in Sheath Taken from Reel Shipped by Rail Across Continent. (High cycle-rate field failure.)

FIG. 17.—Fatigue Cracking in Lead Alloys.

temperature-induced fatigue failure from the field. Both show the same 45-deg cross-hatching of cracks.[11] The field failure differs, however, in that the fatigue cracks contain corrosion products. The fatigue cracks in the lead-1 per cent antimony sheath run through the areas in which extensive precipita-

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[11] Another type of field cracking in which fissures penetrate normal to the sheath surface is not duplicated in flat cantilever-beam type specimens.

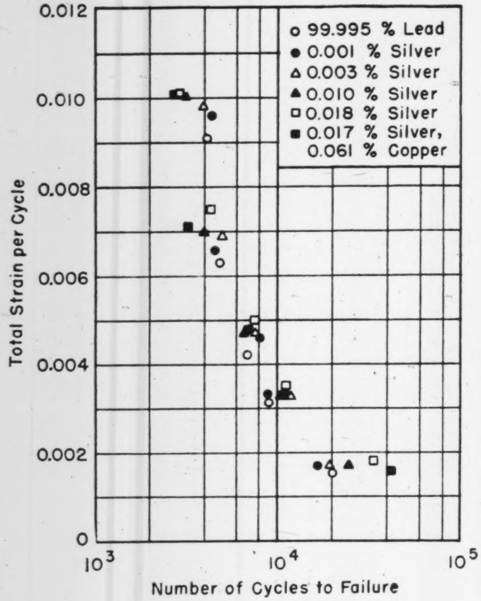

FIG. 18.—Effect of Silver on Cycle Life of Lead Cable Sheath Alloys.
Tested at ¼ cpm, 85 F and zero mean strain.

tion and agglomeration of the antimony-rich phase has occurred. The fatigue failures which took place at high cycling rates are in a lead-0.010 per cent silver alloy. Again the cracks progress through grain boundaries and the laboratory specimen is very much the same in appearance as the field failure.

Despite the similarity of the cracking in laboratory tests and in the field failures in a slow cycle fatigue test, and the close simulation in the design of the laboratory test to field conditions, the life of cable sheath in the field cannot be accurately predicted from laboratory fatigue tests alone. This is readily understood when it is considered that all of the environmental variations are not duplicated in the laboratory test. Since the temperature changes in the field are erratic the strain range does not vary in a simple sinusoidal manner; there is also corrosion (see Fig. 17(*b*))

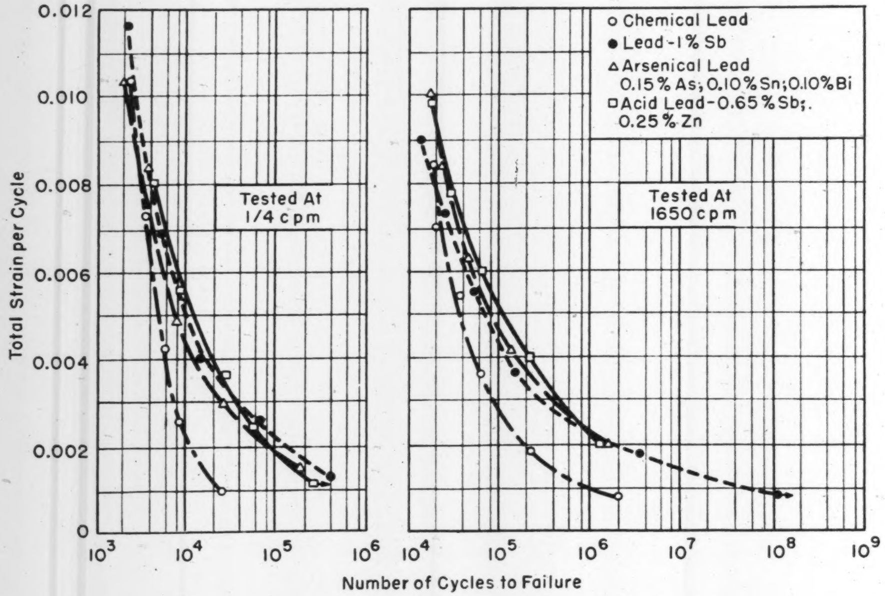

FIG. 19.—Effect of Composition on Cycle Life of Lead Cable Sheath Alloys.
Tested at 85 F and zero mean strain.

and sometimes mechanical damage that in most cases accelerate fatigue cracking. Nevertheless, laboratory fatigue tests are extremely useful in establishing comparative expected field life when the limitations of the tests are adequately appreciated. An example of this usefulness is illustrated in the next section discussing the effects of composition on fatigue life.

Alloy Composition and Cycle Life:

Throughout the years in which cables have been sheathed with lead there has been a continuing search for improved alloys. Improvements can be effected by alloying, by changes in extrusion practice, and by heat treatment. Frequently all three enter into the production of the optimum sheath. Some results of alloying on fatigue properties are shown in Figs. 18 and 19.

The lead alloys in Fig. 18 contain a graded series of silver and copper extending through only a relatively low maximum concentration. The effect of alloying in this range on fatigue properties is small but not negligible; particularly at low strain ranges there is a small improvement in cycle life as a result of adding silver or silver and copper to the lead. In Fig. 19 the concentration range of the alloying elements has been extended to include alloy additions up to about 1 per cent. For comparison, fatigue data are also shown for chemical lead[12]. Results for two different rates of cycling are included. Some features in this comparison are worth emphasizing since they can probably be generalized. At high strain ranges alloying has but a limited effect on cycle life—there seems to be no significant advantage in alloying if high strain ranges are of primary importance. For low strain ranges (below 0.004) that are of primary importance in telephone applications, alloying has a considerable effect in extending the cycle life of cable sheath.

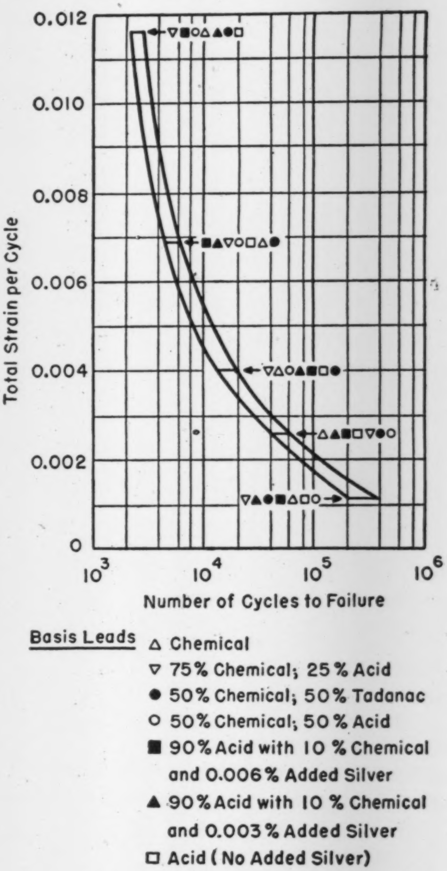

FIG. 20.—Effect of Basis Lead on Cycle Life of Lead - 1 per cent Antimony Cable Sheath Alloys

Tested at 85 F and zero mean strain aged 5 yr. prior to test.

NOTE.—Limiting parameters are shown for seven strain-cycle curves representing tests on sheathing alloys made from the following basis leads. The order of each strain level studied is shown by the sequence of symbols.

The three higher alloyed leads shown in Fig. 19 have very nearly the same fatigue characteristics in both slow and high rates of cycling. One should not infer from this, however, that the quantity of the added element is the only important factor in producing longer fatigue life. There are unquestionably specific effects of different elements for some are in solution, some have only very limited solubility, and others form compounds. There is this sameness, however,—all raise the low recrystallization temperature of lead and minimize recovery processes. This tends to promote retention of whatever work hardening is induced by prior strain cycles. This certainly plays a part in improving fatigue life in low strain ranges.

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[12] Chemical lead is a low alloy lead containing nominally 0.06 per cent copper and 0.020 per cent silver.

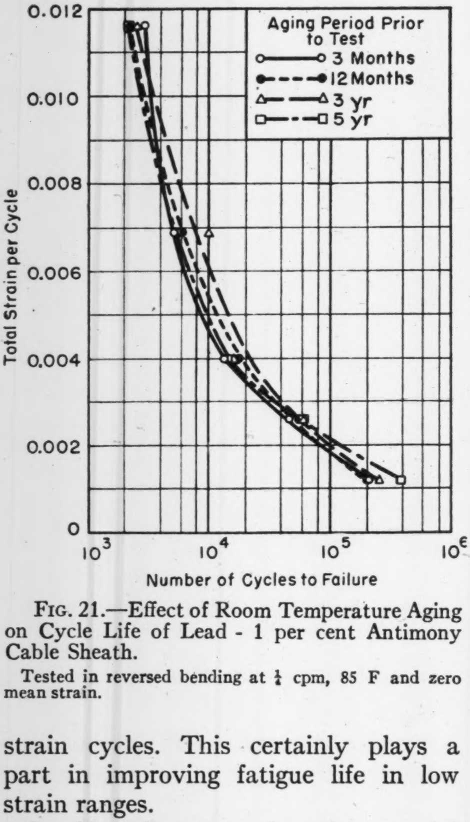

FIG. 21.—Effect of Room Temperature Aging on Cycle Life of Lead - 1 per cent Antimony Cable Sheath.

Tested in reversed bending at ½ cpm, 85 F and zero mean strain.

Rapid cycling tests place the material in the same general order of fatigue life as do the slow cycling tests for the four alloys shown in Fig. 19. Usually this is the case but the degrees of difference may vary with different alloys. An exception is shown in Fig. 7 which was discussed earlier. The occasional exception suggests that high cycle rate tests, if used, are best supplemented by slow cycle rate tests to obtain data for conditions nearer to those in the field.

The effect of minor impurities, as encountered in the usual grades of lead, on the fatigue characteristics of lead-1 per cent antimony alloy are not significant as may be seen from the data shown in Fig. 20. The sheaths were made from chemical lead, acid lead, various mixtures of the two with minor quantities of other elements added, and high-purity lead all alloyed with 1 per cent of antimony. The seven compositions have essentially the same fatigue characteristics.

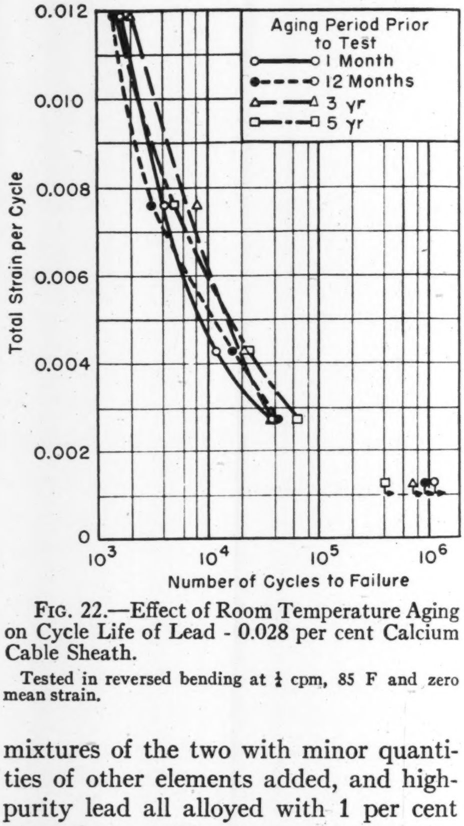

FIG. 22.—Effect of Room Temperature Aging on Cycle Life of Lead - 0.028 per cent Calcium Cable Sheath.

Tested in reversed bending at ½ cpm, 85 F and zero mean strain.

In alloys of the dispersion hardening type, strength properties change with time. Consequently, the question arises, how does the fatigue life change with time? Data for the lead-antimony and for lead-calcium sheaths are shown in

Figs. 21 and 22. It is quite clear that aging has but little effect on cycle life,—at least for these two alloys—despite large changes in other properties. Only in the low strain ranges is any definite trend indicated and the increase in cycle life for the specimens aged longest prior to test is not great enough to be deemed significant. Certainly the aging has not decreased the cycle life!

Summary:

The fatigue testing of lead alloys requires close simulation of service conditions to obtain significant data. To accomplish this, tests are conducted at relatively slow cycle rates (to simulate temperature change effects), at a controlled temperature and under conditions where cycle life can be related to strain rather than stress. High rates of cycling are included when, under service applications or in transportation, low-amplitude, high-cycle rate vibrations may be encountered. For better correlation with field data the laboratory tests are made at a series of strain ranges, one of which encompasses the conditions in service. The effect of temperature on cycle life is useful since alloys are used at temperatures other than the test temperature and because the temperature effect is different for different alloys. The effect of variation in composition is such that only from actual tests can these effects be predicted. Small alloying additions generally have only a minor effect on fatigue life, while larger quantities may markedly improve fatigue life at low strain ranges but not at high strain ranges. Aging prior to test appears to have but little effect on cycle life of age hardening sheath alloys.

Acknowledgment:

Many members of the Bell Telephone Laboratories have contributed to the development of the test methods and the accumulation of test results reported in this paper. Among these are S. M. Arnold, W. Babington, J. R. Boettler, J. P. Guerard, J. P. Ahrens, and K. M. Olsen. The early work was carried out under the direction of J. R. Townsend, the later work under E. E. Schumacher, both of whom have made important contributions to the development. We are also happy to acknowledge the many helpful discussions with G. M. Bouton during the preparation of this paper. He did the development work on the fatigue machine shown in Fig. 3; he has been generous with his experience both in the fatigue testing of lead and in the examination of field failures. G. M. Bouton and E. E. Thomas made available the photomicrographs used. Most of the cables were furnished by J. W. Kennard and his associates and manufactured by the Western Electric Co. The series of lead-silver alloys (20) were furnished by H. S. Phelps of the Philadelphia Electric Co. from pipe samples extruded by the Okonite-Callendar Co.; the arsenical lead alloy (16, 21) was supplied as pipe by L. F. Hickernell of the Anaconda Wire and Cable Co.

REFERENCES

(1) J. R. Townsend, "Fatigue Studies of Cable Sheath Alloys," *Proceedings*, Am. Soc. Testing Mats., Vol. 27, Part II, p. 153 (1927).

(2) S. Beckinsale and H. Waterhouse, "The Deterioration of Lead Cable Sheathing by Cracking," *Metal Industry*, London, Vol. 32, p. 302 (1928).

(3) P. Dunsheath and H. A. Turnstall, "Physical Properties of Lead Cable Sheath," *Journal*, Inst. Electrical Engrs., Vol. 66, p. 280 (1928).

(4) J. Cournot, "Improving the Properties of Lead by Slight Additions of Tin and Cadmium," *Le Genie Civil*, Vol. 92, p. 353 (1928).

(5) O. Haehnel, "Die interkristalline Brüchigkeit des Bleis," *Zeitschrift für Metallkunde*, Vol. 19, p. 492 (1927).

(6) C. W. Dollins, "An Investigation of Creep, Fracture and Bending of Arsenical Alloys for Cable Sheathing," University of Illinois Engineering Experiment Station Bulletin Series No. 394, Vol. 48, p. 60 (1950).

(7) C. S. Schifreen, "Cyclic Movement of Cable—Its Cause and Effects on Cable-Sheath Life," *Transactions*, Am. Inst. Electrical Engrs., Vol. 63, p. 1121 (1944).

(8) H. F. Moore, B. B. Betty and C. W. Dollins, "Creep and Fracture of Lead and Lead Alloys," University of Illinois Engineering Experiment Station Bulletin Series No. 272, Vol. XXXII (1935).

(9) H. F. Moore, B. B. Betty and C. W. Dollins, "Investigation of Creep and Fracture of Lead and Lead Alloys for Cable Sheathing," University of Illinois Engineering Experiment Station Bulletin Series No. 306, Vol. XXXV (1938).

(10) L. G. Ferguson and G. M. Bouton, "Effect of Coating of Polybutene on the Fatigue Properties of Lead Alloys," Symposium on Stress Corrosion Cracking of Metals, p. 103, Am. Inst. Mining and Metallurgical Engrs.-Am. Soc. Testing Mats., (1945). (Symposium issued as separate publication, *STP No.* 64.)

(11) J. F. Eckel, "The Influence of Frequency on the Repeated Bending Life of Acid Lead," See p. 745, this publication.

(12) C. H. Greenall, "Testing Cable Sheath for Fatigue," *Bell Telephone Laboratories Record*, Vol. 13, p. 12 (1934).

(13) H. F. Moore and C. W. Dollins, "Fracture and Ductility of Lead and Lead Alloys for Cable Sheathing," University of Illinois Engineering Experiment Station Bulletin Series No. 347, Vol. 41 (1943).

(14) J. R. Townsend and C. H. Greenall, "Fatigue Studies of Telephone Cable-Sheath Alloys," *Proceedings*, Am. Soc. Testing Mats., Vol. 30, Part II, p. 395 (1930).

(15) D. W. Roper, Discussion on Paper by H. F. Moore and N. J. Alleman, University of Illinois Engineering Experiment Station Bulletin Series No. 243, Vol. 29, p. 5 (1932).

(16) H. Halperin, "Load Ratings of Cable," *Transactions*, Am. Inst. Electrical Engrs., Vol. 58, p. 535 (1939).

(17) E. E. Schumacher, "Communications Metallurgy," *Journal*, Inst. Metals, Vol. 18, p. 1 (1950).

(18) J. C. Chaston, "Properties of Lead and Lead Alloy Cable Sheaths," *Electrical Communication*, Vol. 13, p. 31 (1934).

(19) Report of Committee E-1 on Methods of Testing, Appendix I, "Report of Task Group on Effect of Speed of Testing on Fatigue Test Results," *Proceedings*, Am. Soc. Testing Mats., Vol. 50, p. 421 (1950).

(20) H. S. Phelps, F. Kahn, and W. T. Magee, "Influence of Small Percentages of Silver on the Tensile Strength of Extruded Lead Sheathing," *Proceedings*, Am. Soc. Testing Mats., Vol. 48, p. 815 (1948).

(21) L. F. Hickernell and C. J. Snyder, "F-3 Lead Alloy—An Improved Cable Sheathing," *Transactions*, Am. Inst. Electrical Engrs., Vol. 65, p. 563 (1946).

DISCUSSION

MR. JOHN F. ECKEL[1] (*presented in written form*).—The authors have made an excellent analysis of factors involved in fatigue testing of lead and its alloys when applied to cable sheath. They have also emphasized the desirability of simulating service conditions as closely as practicable and have indicated the differences that generally exist between laboratory tests and service conditions.

In laboratory tests the three fundamental variables are readily controlled, particularly temperature and frequency. Maximum strain per cycle may be more elusive if the laboratory test is made on a cable section. Structural factors in the cable core often introduce strain concentrations that differ from one type of cable to the next. For instance, it was shown by Halperin and Betzer[2] that the same sheath material in a dummy manhole test had a longer bending life on belted cable than on shielded cable.

As suggested in my discussion of their paper, the difference in bending life on the two types of cable must lie in differences in strain concentration factors. As I picture all of these laboratory tests, good correlation can be obtained between them if all three fundamental variables are properly controlled.

In work with power cables, the lead sheath is generally subjected to three types of repeated bending tests. These are:

1. Strip bend test.
2. U-bend test on cable sections.
3. Dummy manhole tests on cable sections.

The strip bend test offers the best method for control of variables, although even here uncertainties may be introduced if there are irregularities in contour, such as tape markings. The U-bend test on cable sections introduces structural variables that must be considered in interpreting results. Although there is a wider spread in test results than with the strip test, the correlation in general is quite good, provided sufficient attention is given to proper determination of the maximum strain per cycle.

Still greater spread in results occurs with the dummy manhole test in which additional variables are introduced. For instance, the strain at any one point on the sheath is not necessarily the same in succeeding cycles. Nevertheless, the dummy manhole test is a valuable test and has been widely utilized.

One statement made by the authors, I believe, is open to question. On page 728 they say "It is probably safe to generalize from this observation that the stiffer the alloy, the less its fatigue life is affected by variations in cycle rate." We have tests running on a number of alloys that are considerably stiffer than any of the grades of commercial leads on which tests have been completed. Preliminary results on tests at frequencies above 24.8 cycles per day indicate larger values for the constant m in the equation

$$\log l = \log b - m \log f$$

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[1] Research Laboratory, General Electric Co., Schenectady, N. Y.

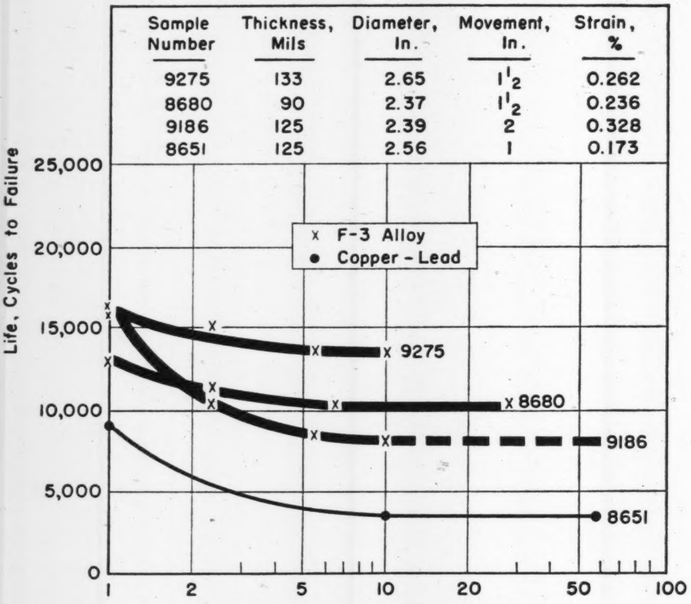
[2] Herman Halperin and C. E. Betzer, "Lead-Alloy Sheaths for Underground Power Cable," *Transactions*, Am. Inst. Electrical Engrs., Vol. 70, Part 1 (1951).

This would mean that these materials are affected more by frequency than the commercial leads. We are not able at this time to state whether the higher *m* values are characteristic of the materials or result from the greater stiffness.

One question I should like to ask concerns Fig. 8. Have the authors any information that might indicate the presence of strain aging and if so the relative amounts in each of the two alloys. I raise this question because I strongly suspect that the increase in creep rate in certain arsenical alloys reported by Halperin and Betzer[2] may be caused by strain aging.

MR. C. J. SNYDER.[3]—The data presented by Messrs. Gohn and Ellis confirm some results, obtained in our laboratory on full-size cables, which we are presenting to the American Institute of Electrical Engineers.[4]

Figure 6 shows that the speed of testing is no longer critical, after it falls below 5 or 10-min cycles. We usually express the testing speed at so many minutes per cycle. Our data, shown in Fig. 23, confirmed this almost exactly, showing no further drop in life as the testing speed was decreased from 10-min to 60-min cycles.



Sample Number	Thickness, Mils	Diameter, In.	Movement, In.	Strain, %
9275	133	2.65	1½	0.262
8680	90	2.37	1½	0.236
9186	125	2.39	2	0.328
8651	125	2.56	1	0.173

FIG. 23.—Bending Life *versus* Testing Speed.

A number of figures in the paper show that lead appears to have some semblance of an endurance limit in fatigue; that is, at relatively high strains, the strain is no longer critical, but as the strain is reduced, a point is reached where the life of the lead specimen picks up rapidly, and at low strains very high values of life are obtained.

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[3] Supervisor, Metallurgical Laboratory, Anaconda Wire & Cable Co., Hastings-on-Hudson, N. Y.

[4] L. F. Hickernell, A. A. Jones, C. J. Snyder, "F-3 Lead Alloy Cable Sheath-Effect of Bending and Creep on Life," *Transactions*, Am. Inst. Electrical Engrs., Vol. 70, Part 1 (1951).

Most of the data reported was for antimonial lead, whereas our experience is mostly with arsenical lead, but the two materials seem to have comparable properties in laboratory testing, even when the antimonial lead is laboratory aged.

However, when applied to electric power cables, subjected to heating and variable loading cycles, the antimonial lead appears to lose some of its good properties with time, whereas the arsenical lead appears to be little affected.

MR. R. L. TEMPLIN.[5]—I should like to ask the authors if it is characteristic to get fatigue failures that are intercrystalline or transcrystalline in this kind of material.

MR. G. M. BOUTON.[6] — At small strains, most of the failures are intercrystalline. At very large strains, the grain deformation and tearing action give failures that go across the grains as well as in between grains.

MR. C. W. DOLLINS[7] (*by letter*).—The authors are to be congratulated for their keen insight of the many variables in the testing of lead and lead alloys, and for having enriched our conception of many of these variables in connection with fatigue studies of these alloys. The authors conclusion that strain, rather than stress, be the criterion in the fatigue testing of lead meets with the approval of most investigators. The authors' suggest that studies of the effect of change in the strain wave shape and rest periods are needed. Such studies have already been undertaken in a small way at the University of Illinois. Interrupted cycles with two rest periods have been used for several years and only recently have tests with sinusoidal cycles been conducted. Enough data have not as yet been collected to tell what extent recovery may have affected the test results in the interrupted strain cycle tests.

It is difficult to compare directly the fatigue test results of the various laboratories testing lead alloys as no two investigators are using the same type specimen. Some control the point of failure by the use of cantilever specimens, some by notched specimens. One laboratory forces the specimen to strain the desired amount by insulated steel guides, while one laboratory, University of Illinois, uses a simple beam type of specimen with a 2-in. gage length that allows considerable choice as to the location of failure in the specimen, which consequently leads to more scatter in the test results. All methods, however, give valuable data for screening alloys.

The authors' conclusion that the effect of variation in cycle rate is less for the "stiffer" alloys is interesting. The more creep-resistant an alloy is the less permanent deformation takes place per cycle, and in the slow rate cycle tests this becomes important. Also of interest is the conclusion that aging does not change to any great extent the cycle life of the 1 per cent antimony and the 0.028 per cent calcium alloys. Limited tests at the University of Illinois have confirmed this in the case of a 0.02 per cent calcium-0.016 per cent magnesium lead alloy.

MESSRS. G. R. GOHN AND W. C. ELLIS (*authors' closure*).—Mr. Eckel has raised a number of important points: The first was the question of whether strain concentration is important in the fatigue test; and, of course, it is. It is a condition that cannot be created in a completely satisfactory way in the laboratory but is present in the field. We think that the presence of such strain concentrations is one reason why life in the field cannot be predicted from laboratory tests. In the laboratory we can only compare alloys and conditioning treatments.

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[6] Member of Technical Staff, Bell Telephone Laboratories, Inc., New York, N. Y.

[7] Engineering Experiment Station, University of Illinois, Urbana, Ill.

The second question concerned the statement that generally we would expect the life of stiffer alloys to be affected less by change in cycle rate. We think that the variance from our conclusion arose because of a difference in definition of life-time. Life-time in our paper is the number of cycles to failure while Mr. Eckel measures life-time in days to failure.

We do not know to what extent strain aging may have entered into the tests, the results for which are shown in Fig. 8, but we do not think that it was an important factor. We did not find for these compositions a significant difference in fatigue life for tests made shortly after extrusion and 5 yr later. We conclude that the relatively large change in amount and precipitation of the second phase over this long period of time had but a small effect on fatigue life. Strain aging effects during test would be expected to have much smaller effects on the amount and distribution of precipitate.

In static tension tests, however, recrystallization during test is known to accelerate creep rate. Presumably other processes involving atomic mobility, such as strain aging, may also influence creep rates, and to the extent that creep enters into the fatigue mechanism, may likewise affect fatigue life. Until we know more about the mechanism of fatigue damage it does not seem likely that we shall be able to explain differences like those shown in Fig. 8.

We are glad to learn that Mr. Snyder has confirmed our findings that the effect on life of changes in cycle rate is not great in the range of $\frac{1}{4}$ cpm. Although Mr. Snyder has correctly interpreted the fatigue curves in pointing out the small effect of changes in strain range at high values and the large effect at low values, there is a hazard in thinking that lead alloys have endurance limits. We prefer to characterize the fatigue endurance of lead alloys in terms of specific values of strain range and corresponding life. This is in line with the findings for most non-ferrous alloys in which there is no true endurance limit.

As pointed out by Mr. Bouton, fatigue failures in lead alloys, from examination after fracture, are generally intercrystalline rather than transcrystalline. In an alloy that may recrystallize under test we cannot be sure, however, that the fatigue crack does not start transcrystalline, as is observed for higher melting point metals. An initially incipient transcrystalline crack, through subsequent structural adjustment, for example recrystallization or grain boundary migration, may have relocated at a grain boundary.

We are pleased that studies have been undertaken by Mr. Dollins on the effects of strain wave shape and rest periods. These are a part of field conditions and information on their effects should aid in correlating field service and laboratory tests.

We are glad that this paper has provoked so much discussion, and to those who have commented we extend our thanks. Their comments have added much to the value of the paper.

THE INFLUENCE OF FREQUENCY ON THE REPEATED BENDING LIFE OF ACID LEAD*

By John F. Eckel[1]

Synopsis

A repeated-bending test for lead strip is described that utilizes a new type of test specimen and a new type of grips. It is shown from test results at 110 F that a definite relationship exists between frequency and the bending life of acid lead between 0.27 per cent and 0.65 per cent maximum strain per cycle and between 6.63 and 7440 cycles per day. Extrapolation gives an indicated bending life at 1 cycle per day that is shorter than that found under service conditions. This discrepancy and differences between laboratory tests and service conditions are discussed. It is suggested that opportunities for recovery existing under service conditions are primarily responsible for a longer life of lead cable sheath than that indicated by laboratory tests.

There are three important variables affecting the life of lead sheath on power cables where such cables are subjected to manhole bending in service. These variables, frequency, maximum strain per cycle, and temperature, have been studied extensively and evaluated under a variety of service conditions. Of these, only frequency approaches a fixed value. Halperin (**1**)[2] and also Schifreen (**2**) have shown that frequency of bending in service is essentially one cycle per day although there are some variations. The magnitude of strain per bending cycle in lead sheath varies according to the installation and load cycles. Studies by Schifreen (**2, 3**) and also by Halperin and Betzer (**4**) have indicated that it is well below 1 per cent, generally ranging from 0.2 to 0.6 per cent per cycle. There are also wide variations in sheath temperature, but according to Halperin and Betzer (**4**) the annual average sheath temperature of a well-loaded cable is about 110 F.

This study was undertaken to establish a more definite relation between bending life of lead and frequency of bending that would permit a justifiable extrapolation to a frequency of one cycle per day. Such an extrapolation, it was felt, would serve as a basis from which estimates could be made of the service life expectancy of lead sheath subjected to manhole bending. It should be reemphasized that lead in service as cable sheath is performing within its creep range and well above its recrystallization temperature. Lead should consequently be considered under these circumstances as a "high-temperature" metal. Furthermore, it should be possible to predict from results obtained on lead the general behavior of other metals when they are subjected to similar repeated strains in their own "high-temperature" range.

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* Presented at the Fifty-fourth Annual Meeting of the Society, June 18–22, 1951.

[1] Research Laboratory, General Electric Co., Schenectady, N. Y.

[2] The boldface numers in parentheses refer to the list of references appended to this paper, see p. 756.

PREVIOUS WORK

Early repeated-bending tests[3] employing lead strips as test samples were generally operated at high frequencies. The first of such tests was described by Jewett (6) in which he superimposed vibrations at a frequency of 108 cycles per sec on strips of lead under tension. He reported no values for the amplitude of vibration either in terms of deflection or strain. Later, Townsend (7) reported a test at the lower frequency of 700 cycles per min in which he used a deflection of 0.11 in. in a length of 3 in. but did not calibrate his test in terms of strain. This deflection according to Townsend "corresponded to a calculated stress of 1350 psi, which was just within the apparent elastic range of most of the experimental alloys investigated." Dunsheath and Tunstall (8), using a test similar to that of Townsend, investigated the effect of variations in the amplitude of movement on the bending life of lead strips. However, they did not correlate their results with any strain measurements.

Moore and Dollins (5) placed repeated-bending tests on a sounder basis when they recognized the need to express the amplitude of bend in terms of strain. They developed a relationship based on the loading of a cantilever beam by which the amount of strain per cycle could be approximated in their tests. By varying the frequency from 1440 to 374,720 cycles per day, they were able to show that fewer cycles were required for fracture at low frequencies than at high frequencies. This was confirmed by Ferguson and Bouton (9) over a range of frequencies from 48 to 2,112,000 cycles per day. Although Ferguson and Bouton did not express their results in terms of strain, there was one important feature of their test that represented a departure from previous tests. They used a test specimen designed to produce maximum strain at the center of a curved section instead of the flat specimens used by previous investigators. Wiseman (10) has also described a strip bending test in which curved sections have been used. Later, Dollins (11) used a test in which the specimen took the form of a simple beam. He developed a method based on the simple beam formula by which he computed the amount of strain to which his samples were subjected in each bending cycle. Dollins employed frequencies as low as 24 cycles per day and showed that still fewer cycles were required to cause fracture at this lower frequency.

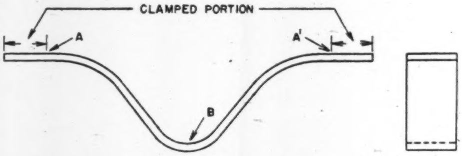

FIG. 1.—Form of Test Specimen with Curved Section Used by Ferguson and Bouton.

The 1950 report of Committee E-1 (15) shows that the fatigue life of other metals is known to be affected by frequency. This is particularly true when tests are made under conditions of stress and temperature that cause creep. However, the frequencies thus far employed are considerably higher than those used by Dollins.

REPEATED-BENDING TEST

An effort was made to utilize the specimen described by Ferguson and Bouton (9) in setting up a repeated-bending test that could use strip samples either cut from cable sheath or extruded in the laboratory. Difficulty was at once encountered in the erratic results caused by varied degrees of cracking at the grips, points A and A' in Fig. 1. In several tests, actual fractures occurred at these points. Various methods were used to relieve strain concentration at points *A* and *A′*, some of which were moderately successful to the extent that all fractures occurred at point *B*. However, severe cracking at the grips was not eliminated and general cracking still occurred over the entire length of the specimen, particularly on high-purity lead and the softer alloys.

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[3] The term "repeated-bending test" has been used previously by Moore and Dollins (5) to designate a type of fatigue test in which the maximum strain per cycle is considered a better criterion of the severity of movement than the stress in the outer fibers.

General cracking of the test specimen was not considered objectionable, but tests on the harder alloys showed a greater tendency for strain to be concentrated at point *B*. This indicated that a satisfactory comparison on all types of materials could be obtained only if the amount of strain at this point of fracture were more nearly equal in all tests. It was therefore considered advisable to modify the test specimen and to use a different type of grip.

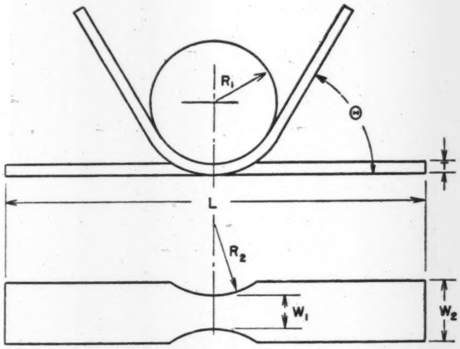

FIG. 2.—Dimensional Variables in Test Specimen with Curved Section.

A study was made of the effects of dimension variables (Fig. 2) in a test specimen with a curved section. This resulted in the adoption of the U-shaped test specimen shown in Fig. 3. Values for dimension *X* depend upon the length of strips *L* (Fig. 3) from which specimens are formed. (Variations in *L* are discussed below in connection with strain measurements.) The notches at the bottom of the U-shaped specimen were introduced so that strain concentration in this region would be comparable in soft and hard lead alloys.

In order to eliminate cracking at the grips, the type of grip shown in Fig. 4 was employed. The principal feature of these grips is the pivoting action that has been obtained by the use of ball bearings. Test specimens are easily inserted into these grips by means of the slots punched in each end of the U-shaped specimen.

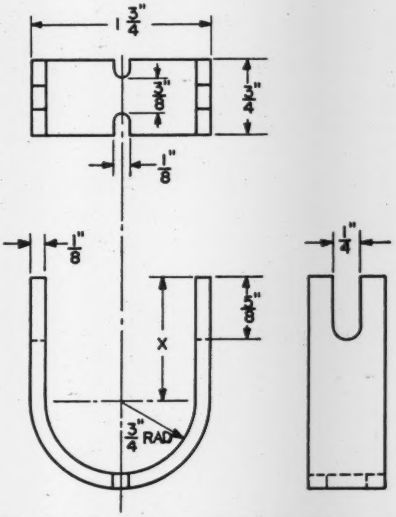

FIG. 3.—Dimensions of U-Shaped Test Specimen.

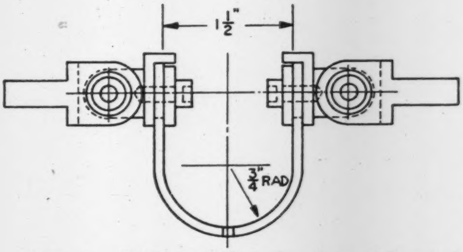

FIG. 4.—U-Shaped Test Specimen Mounted in Pivoted Grips.

The design of the testing machines was made around a unit containing ten test stations. The construction of each unit is indicated in Fig. 5. Reciprocating motion was obtained by means of an adjustable crank that permitted variations in the magnitude of movement. Each unit was so designed that all ten stations operated at the same frequency.

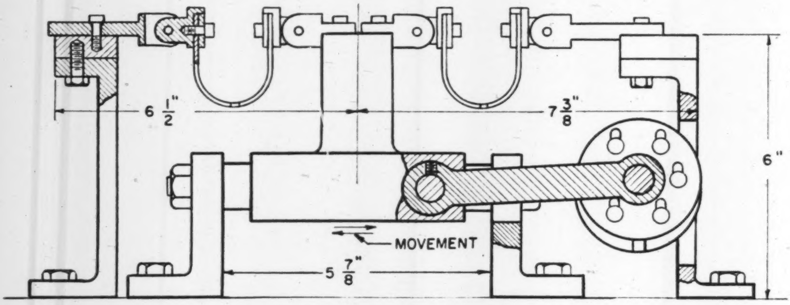

FIG. 5.—Construction of Standard Unit Showing Standard Grips and Specimens in Position for Test.

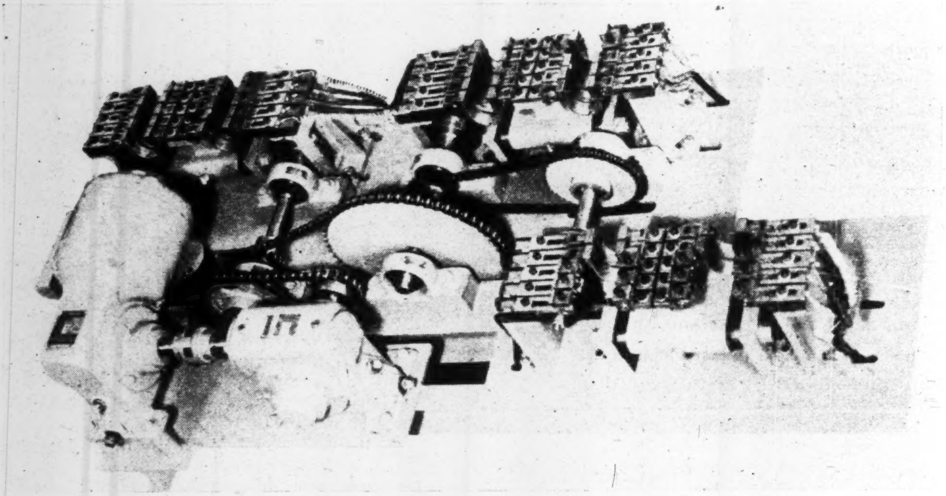

FIG. 6.—Testing Machine With Three Units With Ten Stations Each.

By the use of standard units, a certain amount of flexibility in machine construction was obtained since any desired combination of frequencies was possible by the proper use of gear and sprocket ratios. A photograph of one machine with three of the standard units is shown in Fig. 6. Each of these three units is operated at a different frequency.

In order that testing temperatures could be carefully controlled, all of the units have been placed either in constant temperature rooms or other suitable enclosures.

STRAIN MEASUREMENTS

Reference has already been made to the fact that the distribution of strain in lead specimens was not found to be the same in all alloys and that the difference was most noticeable between hard and soft materials. It was further observed

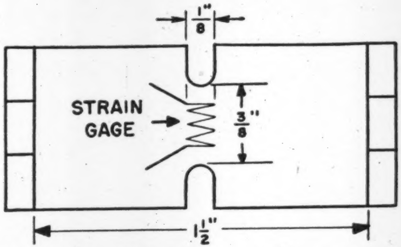

FIG. 7.—Method of Mounting Strain Gage on U-Shaped Test Specimen.

that strain distribution was affected either by thermal or mechanical treatment of any one alloy. Because of these observations, the determination of strain by actual measurement was considered preferable to calculations based upon the shape and movement of the test specimen. SR-4 electrical resistance strain gages with a ⅛-in. gage length have been used for this purpose. They were mounted on the specimens in the manner indicated in Fig. 7.

The nature of movement imposed upon the samples presupposes alternating tension and compression of the same magnitude because of the reciprocating motion produced by a crank turning at constant speed. That this is essentially true is shown by the complete strain cycles in Fig. 8. The amount of divergence from simple harmonic motion is slight as indicated by the dashed curves calculated from

$$S = \frac{S_t - S_c}{2} \sin 2\pi f t \quad \ldots . \quad (1)$$

where:

S = strain at time, t,

f = frequency,

S_t = maximum positive strain from the starting position at the center of bending cycle, and

S_c = maximum negative strain, also from the center of the bending cycle.

S_t and S_c are equal in magnitude and differ only in sign after the first ¼ cycle in any test, the total strain in compression is equal to the algebraic sum of S_t and S_c and the total strain in tension is also equal to this value. That is:

$$S_m = S_t - S_c \ldots\ldots\ldots (2)$$

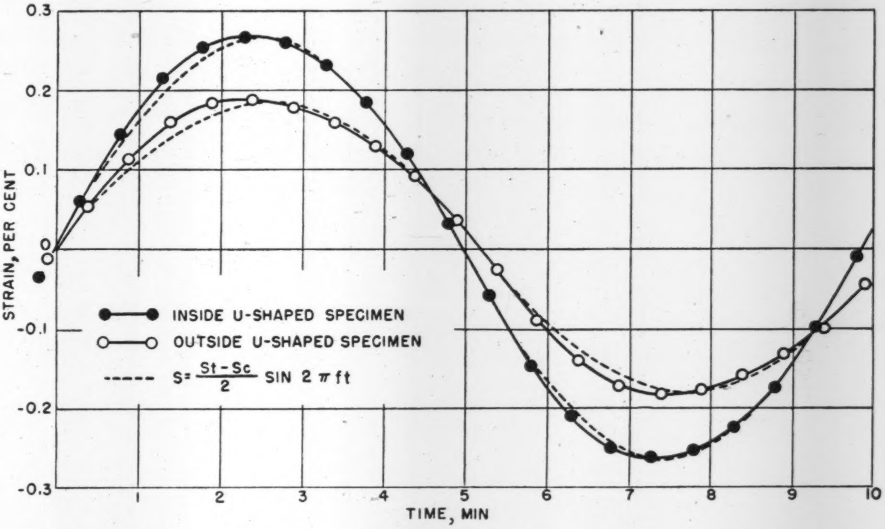

FIG. 8.—Variation in Strain Through One Complete Cycle, Inside and Outside of U-Shaped Specimen of Corroding Lead.

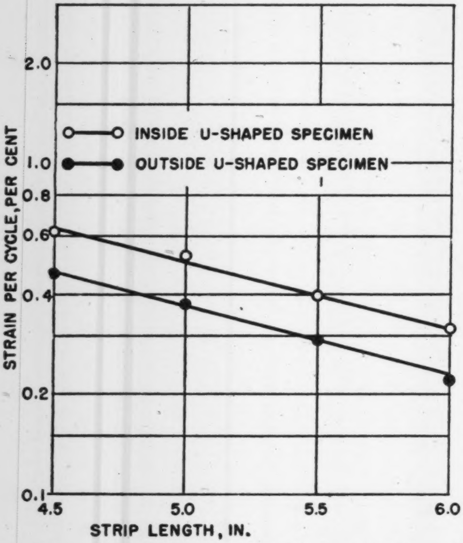

FIG. 9.—Relation Between Strip Length and Strain Per Cycle on Corroding Lead.

where S_m is the total strain per cycle. If measurements are made where maximum strain occurs in the test sample, S_m represents the maximum strain per cycle. The two curves in Fig. 8 illustrate the difference in straining that occurs across the notched section on the inside and outside of the U-shaped specimen. Since maximum straining occurs on the inside of the U, S_m as determined from Fig. 8 is 0.528 per cent per cycle.

Maximum strain per cycle with an amplitude of movement of 0.1 in. can be controlled within the desired range by the length of the strip L from which the specimen is formed, which in turn controls dimension X of Fig. 3. It is therefore necessary to know the relationship that exists between L and S_m. By plotting the logarithm of S_m as a function of L, a straight line was obtained for measurements made on the inside of the U-shaped specimens as well as for measurements made on the outside of the U. This is clearly shown in Fig. 9. A large number of measurements made on several different lead alloys has shown the same relationship to exist in all cases, although the slopes of the resulting straight lines as well as the values of S_m are not always the same. These differences are attributed to the manner in which strain is distributed over test samples of different materials.

In the event that there are differences

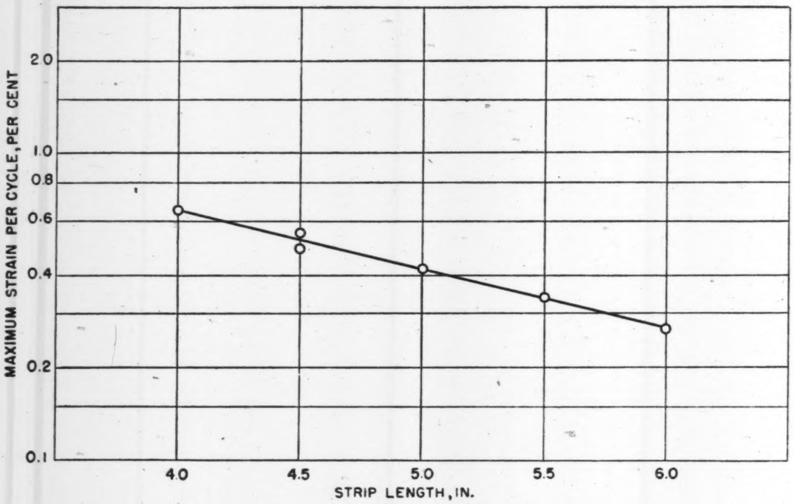

FIG. 10.—Strain Calibration for Acid Lead Specimens Used in Present Investigation.

in thicknesses such as exist in some cable sheaths, it is also necessary to determine the maximum strain per cycle as a function of thickness. It has been found experimentally that S_m is a linear function of T.

With the relationships known that exist between L and S_m and also between T and S_m, it is a simple matter to determine the strains that will be imposed on an alloy with any thermal or mechanical treatment. Since $S_m = 0$ when $T = 0$, it is only necessary to make two measurements regardless of variations in T.

TEST RESULTS ON ACID LEAD

Acid lead containing 0.057 per cent copper and 0.005 per cent bismuth was selected for tests to establish the relationship between bending life, maximum strain per cycle, and frequency at a constant temperature of 110 F. All samples used for these experiments were extruded in the laboratory into $\frac{1}{8}$-by $\frac{3}{4}$-in. strips and were stored 1 yr at 110 F before

TABLE I.—BENDING LIFE DATA FOR ACID LEAD.

Maximum Strain per Cycle, per cent	Cycles to Failure	Average Cycles to Failure	Average Bending Life, days	Maximum Strain per Cycle, per cent	Cycles to Failure	Average Cycles to Failure	Average Bending Life, days
6.63 CYCLES PER DAY				297.6 CYCLES PER DAY			
0.27	1 319			0.27	4 170		
	1 498	1 408	212.4		5 300	4 735	15.9
0.34	1 213			0.34	4 200		
	1 478	1 320	199.1		4 650	4 425	14.9
0.42	1 045			0.42	3 920		
	1 307	1 176	177.4		3 970	3 945	13.3
0.52	1 120			0.52	3 720		
	1 135	1 127	170.0		4 600	4 160	14.0
0.65	1 100			0.65	3 480		
	1 140	1 120	168.9		3 560	3 520	11.8
24.8 CYCLES PER DAY				1488 CYCLES PER DAY			
0.27	1 910			0.27	9 480		
	1 930	1 920	77.4		10 320	9 900	6.6
0.34	1 750			0.34	8 340		
	2 150	1 950	78.4		8 840	8 590	5.8
0.42	1 740			0.42	7 330		
	1 800	1 770	71.4		8 020	7 675	5.2
0.52	1 550			0.52	6 240		
	1 800	1 675	67.5		7 500	6 870	4.6
0.65	1 670			0.65	5 565		
	1 740	1 705	68.6		5 580	5 572	3.8
148.8 CYCLES PER DAY				7440 CYCLES PER DAY			
0.27	3 870			0.27	13 840		
	4 330	4 100	27.6		16 500	15 170	2.0
0.34	4 120			0.34	12 900		
	3 270	3 695	24.8		15 600	14 250	1.9
0.42	3 180			0.42	11 180		
	3 420	3 300	22.1		12 000	11 590	1.6
0.52	2 975			0.52	10 700		
	3 420	3 197	21.5		10 970	10 885	1.5
0.65	2 975			0.65	10 340		
	3 240	3 108	20.9		8 630	10 050	1.4

any of the tests were started. Duplicate tests were made for each combination of strain and frequency employed. A strain calibration (Fig. 10) of the extruded (and aged) strips of the material was made at the time tests were started.

Results of the experiments with acid lead (Table I) can be plotted in several ways. Figure 11 is the conventional type of fatigue data plot for all six frequencies investigated. It differs only in that strain has been used as the ordinate instead of

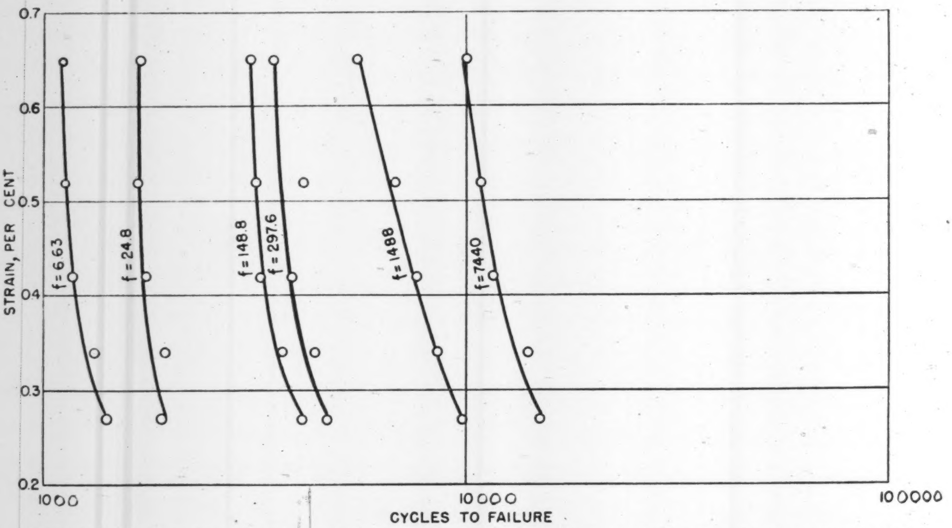

FIG. 11.—Influence of Strain and Frequency on the Number of Cycles Required to Cause Failure in Acid Lead.

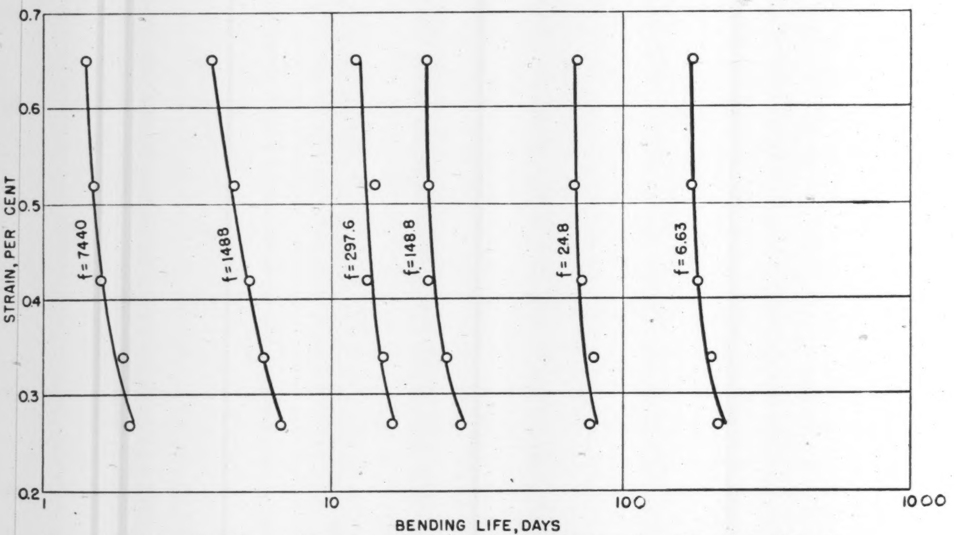

FIG. 12.—Influence of Strain and Frequency on the Bending Life of Acid Lead.

stress. This type of plot, however, tends to distort the picture when bending life is a function of frequency as well as a function of strain. For instance, it may appear at first from Fig. 11 that the resistance to repeated bending is less at low frequencies than at high frequencies. In terms of the number of cycles required to cause failure, this is true, but in terms of the length of time required to cause failure, it does not hold. This is clearly shown by a replot of the data in Fig. 12 in which bending life in days has been used as the abscissa.

That the maximum strain per cycle has a much larger effect upon bending life at the higher frequencies is clearly shown by the curves in Figs. 11 and 12. The slopes of these curves indicate that at the higher frequencies, increased strain per cycle produces a more rapid decrease in bending life over the entire range of strain investigated. It is evident from the curves that the range of strain investigated is well above the endurance limit of acid lead. At lower strains, it is expected that all of the curves would turn more sharply to the right, thus indicating an endurance limit.

The observation of previous investigators, notably Dollins (11), that fewer cycles are required to produce fracture at the lower frequencies is confirmed by the data in Table I. However, from the present data it is possible to establish a more definite relationship between bending life and frequency. By plotting $\log l$ as a function of $\log f$ at constant strain, a straight line results. (See Fig. 13). The general equation of this straight line is

$$\log l = \log b - m \log f \ldots\ldots (3)$$

where l is the bending life in days at frequency f, in cycles per day, b is the bending life (or the number of cycles to fail-

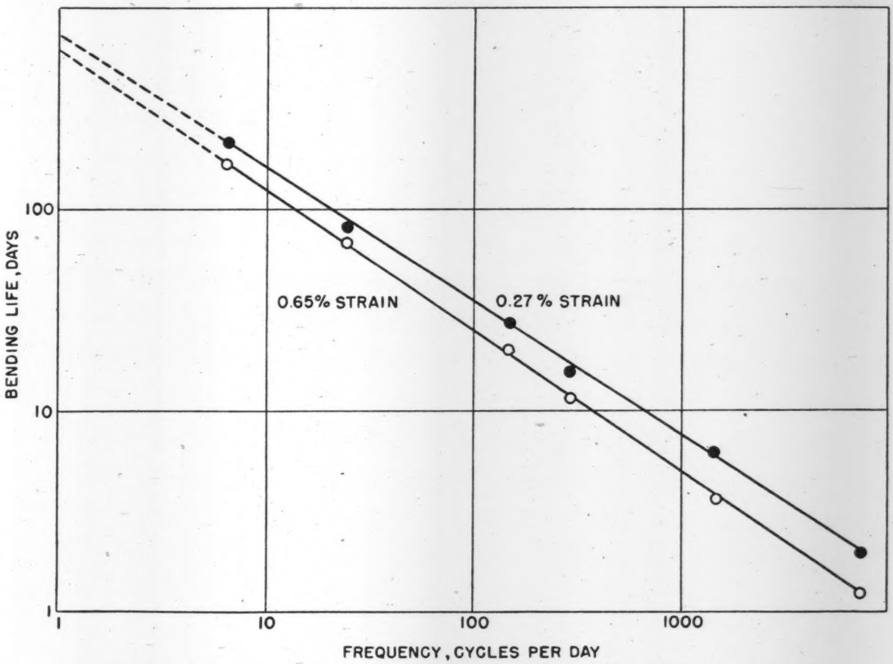

FIG. 13.—Relation Between Frequency and the Bending Life of Acid Lead.

ure) when $f = 1$ and m is the slope of the straight line. Slope m expresses the decrease in bending life with increase in frequency. The experimental values of m found from Fig. 13 are 0.66 when $S_m =$ 0.27 per cent and 0.70 when $S_m = 0.65$ per cent, or in each case approximately $\frac{2}{3}$.

If the number of cycles required to cause fracture (N) is to be considered the criterion of bending life, then the following equation applies:

$$\log N = \log b + n \log f \ldots\ldots (4)$$

It follows that

$$m + n = 1 \ldots\ldots\ldots\ldots (5)$$

DISCUSSION

One of the principal advantages in plotting repeated bending data on lead in the manner just described is that it permits extrapolation to a frequency of one cycle per day with some justification. It is, of course, necessary to assume that no change in the relationship between bending life and frequency occurs over the range of extrapolation. Such an extrapolation has been made in Fig. 13, using data from tests made at two constant strain values. From these extrapolations, values for b were found to be 742 days at a strain of 0.27 per cent and 630 days at a strain of 0.65 per cent.

Under actual service conditions, unalloyed lead sheath has been shown by Halperin (1) to withstand manhole bending for longer periods of time than those indicated by the above values extrapolated to a frequency of one cycle per day. This discrepancy is not surprising when the differences between manhole bending in service and the laboratory test described above are examined. Differences are found in each of the three principal variables: temperature, strain, and frequency. These variables were more closely controlled in the laboratory test than is possible in service. Consequently, the laboratory bending cycle is less complex because temperature, strain and frequency were each fixed for any one test.

Temperature changes resulting from load variations in cables are recognized as the direct cause of manhole bending. If the load were continuously changing, a bending cycle similar to the laboratory bending cycle could be expected except for the continuously changing temperature. This, however, is not the case. As shown by Halperin (1), there is a definite time lag between movement and temperature. In addition, load changes are not continuous and vary from day to day. The resulting bending cycle, although essentially one cycle per day, is discontinuous and affords considerable periods of time in which no movement occurs. Furthermore, as shown by Halperin (1) and also by Schifreen (2), there may be little or no movement one day each week. Such periods in which no movement occurs afford ample opportunity for some recovery to occur in lead cable sheath. This, it is believed, is a very important contributing factor in prolonging the bending life of cable sheath in service. Another important difference between the laboratory test and manhole bending is the amount of strain imposed per cycle. Strain measurements on specimens in the laboratory test have shown that there is no change in the amount of strain per cycle for at least 200 cycles. This condition, it is believed, continues until cracks are first formed. It has been shown by Halperin and Betzer (4) that at any one point on cable sheath in a dummy manhole test, the amount of strain per cycle is not necessarily constant throughout the test. This condition probably accounts in part for the longer life of cables in service.

It is of particular interest that all of the fractures of the acid lead samples tested were intergranular in character. This was expected in light of the observations of Archbutt (12) and subsequent investigators. However, with certain known facts about other metals, it forms the basis from which speculations can be made on their possible behavior in their own "high temperature" ranges.

Intergranular fractures are not uncommon in other metals. This was demonstrated early by Rosenhain (13) and his co-workers who not only observed such fractures in several metals, but also reported grain boundary movement and separation of the type observed by Moore, Betty, and Dollins (14) in lead. These fractures were all caused by the application of long time steady loads in temperature ranges that must be considered as "high temperatures" for the respective metals. Such known facts lead to two questions. First: will intergranular fractures also occur in metals other than lead if subjected to repeated bending of low frequency in their own respective "high-temperature" ranges? Second: will repeated-bending life of other metals be affected by frequency in the same manner as that of lead, thus following the relationship set forth in Eq 3 or another equation similar to it? It seems reasonable to believe that when complete experimental data are available to answer these questions, both answers will be in the affirmative.

SUMMARY

1. A repeated-bending test for lead strip has been described embodying a new type of test specimen and a new type of grips. The test permits close control of the three principal variables affecting the bending life of lead and its alloys. Electrical resistance strain gages have been used to determine the maximum strain per cycle.

2. The influence of strain and frequency on the bending life of laboratory extruded acid lead at 110 F has been determined between strain limits of 0.27 per cent and 0.65 per cent, and bending frequencies of 6.63 and 7440 cycles per day. From the data obtained, the relationship between bending life and frequency has been found to follow the general equation

$$\log l = \log b - m \log f$$

3. The bending life of laboratory extruded acid lead has been determined at a frequency of one cycle per day by extrapolation. These extrapolated values are less than the actual life of unalloyed lead sheath on cables in service. It is suggested that the irregularities in manhole bending which permit both variations in the amount of strain and opportunity for recovery in the sheath during periods of no movement are responsible for the longer life of cable sheath than that indicated by the extrapolated values.

4. It is suggested that the repeated-bending life of metals other than lead at "high temperatures" will be affected by frequency in much the same manner, perhaps following the same general law as that for lead.

Acknowledgment:

The author's thanks are due to L. Zickrick who aided in the development of the test specimen and the testing machines, to S. J. Rush for his part in designing the testing machines, and to L. J. Hartford who made the strain measurements and conducted most of the repeated bending tests.

REFERENCES

(1) H. Halperin, "Load Ratings of Cables," *Transactions*, Am. Inst. Electrical Engrs., Vol. 58, p. 535 (1939).

(2) C. S. Schifreen, "Cyclic Movement of Cable—Its Causes and Effects on Cable Sheath Life," *Ibid*, Vol. 63, p. 1121 (1944).

(3) C. S. Schifreen, "Cable Movement and Sheath-Life Data and their Application in the Design of Cable Manholes." Paper presented at Meeting of Electrical Engineers Transmission and Distribution Committee and Association of Edison Illuminating Companies Cable Engineering Section, Chicago, May 7, 1946.

(4) Herman Halperin and C. E. Betzer, "Studies of Cable Movement and Sheath Life," *Ibid*, May 7, 1946.

(5) H. F. Moore and C. W. Dollins, "Fracture and Ductility of Lead and Lead Alloys for Cable Sheathing," *Bulletin*, University of Illinois, Engineering Experiment Station, Vol. 41, No. 9 (1943).

(6) F. B. Jewett, "The Modern Telephone Cable," *Transactions*, Am. Inst. Electrical Engrs., Vol. 28, Part 2, p. 1079 (1909).

(7) J. R. Townsend, "Fatigue Studies of Telephone Cable Sheath Alloys," *Proceedings*, Am. Soc. Testing Mats., Vol. 27, Part II, p. 153 (1927).

(8) P. Dunsheath and H. A. Tunstall, "The Physical Properties of Lead Cable-Sheaths," *Journal*, Inst. Electrical Engrs., Vol. 66, p. 280 (1928).

(9) L. Ferguson and G. M. Bouton, "The Effect of a Coating of Polybutene on the Fatigue Properties of Lead Alloys," p. 473, Symposium on Stress Corrosion Cracking of Metals, Am. Soc. Testing Mats. and Am. Inst. Mining and Metallurgical Engrs. (1944).

(10) R. J. Wiseman, Discussion of Paper by C. S. Schifreen, *Transactions*, Am. Inst. Electrical Engrs., Vol. 63, p. 1395 (1944).

(11) C. W. Dollins, "An Investigation of Creep, Fracture and Bending of Lead and Lead Alloys for Cable Sheathing," *Bulletin*, University of Illinois, Engineering Experiment Station, Vol. 45, No. 65 (1948).

(12) L. Archbutt, "Failure of Lead Sheathing of Electric Cables," *Transactions*, Faraday Soc., Vol. 17, p. 22 (1921–1922).

(13) Walter Rosenhain and Donald Ewen, "The Intercrystalline Cohesion of Metals," *Journal*, Inst. Metals, Vol. 10, p. 119 (1913).
see also:
Walter Rosenhain and J. C. W. Humfrey, "The Tenacity, Deformation, and Fracture of Soft Steel at High Temperatures," *Journal*, Iron and Steel Inst., Vol. 87, p. 219 (1913).
Walter Rosenhain and Sidney L. Archbutt, "On the Intercrystalline Fracture of Metals under Prolonged Application of Stress," *Proceedings* Royal Soc., London, Vol. A96, p. 55 (1919).

(14) H. F. Moore, B. B. Betty and C. W, Dollins, "Investigation of Creep and Fracture of Lead and Lead Alloys for Cable Sheathing," *Bulletin*, University of Illinois, Engineering Experiment Station, Vol. 35, No. 102 (1938).

(15) H. F. Moore, "Report of the Task Group on Effect of Speed of Testing on Fatigue Test Results," Appendix 1, 1950 Report of Committee E-1 on Methods of Testing, *Proceedings*, Am. Soc. Testing Mats., Vol. 50, p. 421 (1950).

DISCUSSION

Mr. W. C. Ellis[1] (*presented in written form*).—Mr. Eckel, from these very careful measurements of the effect of frequency of straining on the life of acid lead, has made important contributions to the knowledge of the fatigue behavior of lead alloys. We concur with Mr. Eckel that life time—even at one cycle per day—is a number that cannot be carried over directly into a prediction of sheath life in the field. There are too many variables in the field not duplicated in the laboratory to allow for so simple and useful a conclusion.

The value of such tests is rather to compare the relative merits of various alloys as sheath. Not all lead materials will have the same response to a change in the frequency of straining. In general the stiffer alloys—those with a higher level of quasi-elastic behavior—are influenced to a lesser degree by the speed effect. As a consequence, if comparison fatigue tests are made at a high strain frequency, the superiority of a stiffer alloy over unalloyed, or low-alloyed, lead will be less pronounced. These comments are for results obtained with strain ranges reasonably to be expected in field service. We shall look forward to additional valuable results by the author if he extends his studies to alloyed leads.

There are fundamental reasons for the large effect of strain frequency in testing lead and its alloys or, for that matter, any metal at a corresponding temperature. Basically, the rate at which the strain is imposed is important; this permits two results: a variable time for recovery and different flow processes. The two are not unrelated. It is recognized[2] that the mechanism of flow depends importantly on the strain rate and temperature. With decrease in strain rate or rise in temperature, flow changes from "classical" slip by small units to larger units or cells; in the extreme case of cell flow the entire grain, through adjustments by single atoms near the boundary, becomes the unit. This is consistent with observations of grain rotations and boundary movements in creep tests on lead at slow strain rates.

This latter, the diffusive type of flow concentrated near the boundaries, is preferred at slow strain rates and high temperatures because it is the one that can under these conditions change shape most economically as to energy. It is essentially self diffusion, a process favored by temperature and time. Failure by grain boundary separation (whether by creep or by fatigue) may at first quite probably be a segregation of lattice vacancies or voids concentrated by diffusive readjustment under stress. Failure under cyclic strain occurs sooner when this is the primary process than when under high strain rates "classical" slip utilizes the entire grain for shape change.

These considerations seem to explain adequately the broad features of the effect of changing strain rate. Likewise, the confusing effects of rest periods, relief-annealing, under-stressing, and grain size, ought ultimately to be rationalized in terms of flow and recovery processes.

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[1] Member of Technical Staff, Bell Telephone Laboratories, Inc., New York, N. Y.

[2] W. A. Wood, G. R. Wilms, and W. A. Rachinger, "Three Basic Stages in the Mechanism of Deformation of Metals at Different Temperatures, and Strain Rates," *Journal*, Institute of Metals, Vol. 79, p. 159 (1951).

MR. P. R. TOOLIN.[3]—Mr. Eckel has mentioned the possible similarity of room-temperature fatigue tests on lead and high-temperature fatigue tests on other metals. The repeated bending fatigue tests at both 300 and 7200 cpm, which we have made on the alloy Nimonic-80 (75 per cent, Ni, 20 per cent Cr, 2.3 per cent Ti, 0.03 per cent C) at 1200 F may therefore be of interest. At the high nominal-stress end of the *S-N* diagrams for these tests, a specimen tested at 300 cpm fails at fewer cycles but at greater elapsed time than one tested at 7200 cpm. However, at the low nominal-stress region, where the fatigue curves become flatter, the life of a 300-cpm test is less both on a cyclic and on an elapsed time basis than that of one run at 7200 cpm. This latter region is perhaps at a lower portion of the fatigue curve than that explored for lead by Mr. Eckel. At both 7200 cpm and 300 cpm, the fatigue fractures appear to progress in a transcrystalline manner.

It should be noted that, compared to room temperature for lead, 1200 F is a relatively cold temperature for Nimonic-80. Consequently, similar studies at higher temperatures and at both slower and faster testing speeds are of interest. However, such tests on commercial high-temperature alloys would be plagued by metallurgical instability and, if run in air, by oxidation.

MR. A. I. BLANK.[4]—Although the behavior of lead at or near room temperature may be analogous to that of other materials at elevated temperatures, it seems advisable to consider the effect of oxidation, particularly internal oxidation, on the physical properties of those materials at elevated temperature. None of the previous discussion has included any reference to the effect of oxidation. Presumably the data mentioned in the preceding discussion applied to tests at 1200 F. It would seem reasonable to expect that the effect of oxidation at this temperature was considerably greater at the slower speed of testing since the elapsed time per cycle was longer than at the higher testing speed. The effect of oxidation when materials are tested in air at elevated temperatures is quite important and should not be overlooked in evaluating the effect of frequency on fatigue life.

MR. T. J. DOLAN.[5]—In presenting this paper, the author mentioned the discrepancy between field performance and results of the laboratory test. I wonder if some of this difference could be attributed to the specimen design. As I understand it, a cold-forming operation in bending is required which may result in a state of plastic deformation and residual stress different from that in the cable. It also appears possible that the notches in the sides of the specimen at the test section might result in some stress concentrations on straightening which would shorten the life of the laboratory specimen.

MR. C. J. SNYDER.[6]—The data on the effect of the speed of testing presented by Mr. Eckel, are at variance with our data[7] which shows no additional reduction in life at testing speeds below about 10-min cycles.

Our data covers both acid lead and arsenical lead. The specimens were full-size cables. We used strains of 0.23 to 0.33 per cent on arsenical lead, and 0.17 per cent on copper lead.

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[3] Research Engineer, Westinghouse Electric Corp., East Pittsburgh, Pa.

[4] Assistant Research Metallurgist, Chase Brass & Copper Co., Waterbury, Conn.

[5] Research Professor of Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

[6] Supervisor, Metallurgical Laboratory, Anaconda Wire & Cable Co., Hastings-on-Hudson, N. Y.

[7] L. F. Hickernell, A. A. Jones, C. J. Snyder, "F-3 Lead Alloy Cable Sheath-Effect of Bending and Creep on Life," *Transactions*, Am. Inst. Electrical Engrs., Vol. 70, Part 1 (1951).

The strains used by Mr. Eckel are above any semblance of an endurance limit for copper lead and above those permissible in power cable service. It would be interesting to know if Mr. Eckel would obtain a different correlation at strains below those reported.

If lower strains still give the same picture, perhaps the confined area of strain in Mr. Eckel's test specimen does not give a condition similar to that obtained in cable sheath, where the area of strain is spread over a wide area, over several feet of cable.

MR. C. W. DOLLINS[8] (*by letter*).—Mr. Eckel is to be congratulated for an interesting paper describing a new type of bend testing machine for testing lead and lead alloys. The notched specimen used in this machine definitely locates the point of failure. This is an advantage in obtaining consistent data when testing fine-grained lead or lead alloys. However, more scatter in the data is expected when testing the more creep-resistant larger-grained leads. This fact is minimized somewhat as the alloys now used for cable sheathing are usually finer grained than the acid lead reported in these tests where very consistent test results were obtained.

These testing machines are easily loaded with specimens. The specimens themselves are simple to produce, especially when laboratory extruded ribbons are used. Since the strain in the test specimen is limited to the notched section, little increase in specimen length occurs, which, if it did occur, would result in increased strain as the test progresses as in specimens of longer critical sections. The sinusoidal strain pattern is very similar to the stress pattern used in most fatigue testing. The sheathing of power cables in service receives an interrupted strain cycle where, as Mr. Eckel points out, there is time for considerable recovery between peak strain cycles. It has been known for some time that rest periods greatly reduce the creep of lead and lead alloys subjected to steady stress for equivalent times under load.

Mr. Eckel's bending machines are excellent ones to screen the lead alloys as to bending resistance. Since some of his machines have been converted to an interrupted strain cycle, a report on such tests would be of interest. The test results of repeated bending of lead alloys at the University of Illinois where an interrupted cycle is used do not show as great a reduction in the number of cycles necessary for failure at low rates of strain (1 hr) as do Mr. Eckel's tests. It is believed that recovery is the main cause of this.

The author's conclusion that the maximum strain per cycle has a much larger effect upon bending life at the higher frequencies as shown by his curves in Figs. 11 and 12 is of interest. This indicates that as the frequency of straining is increased, more fatigue damage per cycle is done to the material and less creep damage. More slip within the grains may be obtained in the high strain tests, thus weakening the material near the grain boundaries (dislocations).

MR. J. F. ECKEL (*author's closure*).—I think the explanation by Mr. Ellis, of the reason why there is a difference between bending life, as a function of frequency, is very interesting.

It is a little difficult to tie down the strain rate in this type of test, because there is a variable strain rate throughout the cycle, going from zero to the maximum value which depends not only on frequency but also upon total maximum strain per cycle. Furthermore, as I have shown by the curves from Schifreen's paper, maximum strain rates in service do not correspond to the service frequency. The observations of Wood, Wilms, and Rachinger were based upon constant strain rates.

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[8] Engineering Experiment Station, University of Illinois, Urbana, Ill.

Mr. Toolin's data are very interesting and show that information obtained on lead are indicative of the behavior of other metals in their own high-temperature range.

One thing that we might expect, if such tests are carried out at higher temperatures, is that it may be desirable to express results in strain rather than in stress, in order to obtain a good clear picture.

Mr. Blank has pointed out the possible effect of surface oxidation. With lead, that has been covered by a number of previous papers, one in particular by Ferguson and Bouton. They demonstrated that by coating lead samples, the bending life was materially increased. Possibly that is because the rate of oxidation is reduced. We have observed that any alloy which does not oxidize rapidly in our tests will have a much longer bending life. We have not attempted to correlate the rates of oxidation with the test.

Mr. Dolan raised the usual question concerning cold forming. First, let me reemphasize that ambient temperatures are high temperatures for lead. The cable, when installed in a manhole and pulled into ducts, reeled and unreeled, is deformed considerably. In some cases the deformation may be as high as 6 per cent during installation.

In the literature on testing of lead there has been quite a controversy over that one point, whether the characteristics of straightening, for instance, a strip cut from a lead sheath pipe will affect its creep properties. We have to do it anyway, so we just put our tongue in our cheek and go ahead.

We have obtained excellent correlation between our strip test results and U-bend test results made on cable sections.

As I pointed out in my presentation, we do not get quite as good correlation with the dummy manhole test, because there are a large number of other variables thrown into that test.

The notches in the U-shaped specimen were made after it had been completely formed. Hence, there is no straightening after the notches are made as implied by Mr. Dolan.

Mr. Snyder made the statement that beyond the cycle time of 10 min there was practically no change in the life of the arsenical lead alloy. We have been running, for quite some time, tests on that very same alloy, and we have preliminary data at the higher strain values. They, of course, are the ones to break first. Our tests show that these data line up on the same kind of straight line as they do for the acid lead, chemical lead, and common lead which I mentioned before, but different values of constants *b* and *m* are indicated. In fact, preliminary data indicate that constants *b* and *m* may be useful as a means of evaluation of lead alloys. The lower strain correlation is about the same, although there is a large spread of results at these lower values of strain.

The remarks by Mr. Dollins are greatly appreciated, and it is particularly gratifying to know that he concurs in most of the statements I have made.

COMPRESSION TESTS ON LEAD ALLOYS AT EXTRUSION TEMPERATURES*

By G. M. Bouton[1] and G. S. Phipps[1]

Synopsis

Load-deflection measurements made during compression tests on lead and lead-alloy cylinders at various temperatures show the effects of alloying ingredients on the force required to produce deformation. The curves also furnish clues as to changes taking place in the materials during the course of the test. The load, P, to produce definite small deformation in pure lead at various temperatures, T, are shown to follow the relationship $P = Ae^{-BT}$, where A and B are constants for the material. This is the same relationship found by others in extrusion studies. The elements added to lead were those most commonly used in the manufacture of cable sheath, namely, antimony, arsenic, bismuth, silver, tellurium, and tin. The results show that the stronger alloys now used for cable sheathing deform less readily at extrusion temperatures than pure lead or the weaker alloys.

The effect of alloying elements on the plasticity of lead is of importance in the extrusion of cable sheath. Studies of extrudability have been made by several workers,[2] and studies of compression behavior have been made by Zickrick.[3] Direct extrusion studies are complicated by many factors such as friction between the piston and cylinder of the extrusion press and between the lead alloy and surfaces of the extrusion chamber, particularly near the exit orifice. Also, the energy generated by deformation during extrusion causes temperature increases during the test unless very slow extrusion speeds are used. This is particularly true in the region of the die where variations as great as 30 C have been measured during extrusion. In addition, variations in pressure are associated with the decreasing height of the charge during direct extrusion.

The parallel plate plastometer method of measuring plasticity overcomes some of the objections to direct extrusion studies but is itself open to the objection that the distortion of the test specimens during compression makes direct correlation of stress and strain extremely difficult. However, the specimens appear to deform in comparable manners, which should permit direct comparison between load-deformation curves for various alloys and conditions. Also, much valuable information is obtained in the early stages of deformation before the effects of distortion are significant.

In selecting the alloys for test, consideration was given to the elements most commonly used in making com-

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* Presented at the Fifty-fourth Annual Meeting of the Society, June 18–22, 1951.

[1] Member of Technical Staff, Bell Telephone Laboratories, Inc., New York, N. Y.

[2] C. E. Pearson and J. A. Smythe, "The Influence of Pressure and Temperature on the Extrusion of Metals," *Journal*, Inst. Metals, Vol. 45, p. 345 (1931).

[3] L. Zickrick, "Effect of Small Percentages of Certain Metals upon the Compressibility of Lead at Elevated Temperatures," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 99, p. 345 (1932).

TABLE I.—MATERIALS USED FOR COMPRESSION TESTS.

Type of Lead	Composition, per cent					
	Silver	Copper	Bismuth	Zinc	Antimony	Tin
Corroding lead *A*	0.0004	0.0012	0.004	0.0002	0.004	0.0002
Corroding lead *B*	<0.001	<0.001	0.005	......	0.001	......
Corroding lead *C*	0.0002	0.0003	<0.001	0.0002	<0.0003	0.00005
Chemical lead	0.007	0.06	<0.001	<0.001	0.001	<0.001

Lead *A* + 0.16 per cent Antimony
Lead *A* + 0.24 per cent Antimony
Lead *A* + 0.66 per cent Antimony

Chemical Lead + 0.16 per cent Antimony
Chemical Lead + 0.32 per cent Antimony
Chemical Lead + 0.71 per cent Antimony
Chemical Lead + 1.0 per cent Antimony

Lead *A* + 0.1 per cent Bismuth
Lead *A* + 0.1 per cent Tin
Lead *A* + 0.14 per cent Arsenic
Lead *A* + 0.14 per cent Arsenic + 0.1 per cent Bismuth
Lead *A* + 0.14 per cent Arsenic + 0.1 per cent Tin
Lead *A* + 0.14 per cent Arsenic + 0.1 per cent Tin + 0.1 per cent Bismuth

Lead *C* + 0.01 per cent Silver
Lead *B* + 0.06 per cent Tellurium

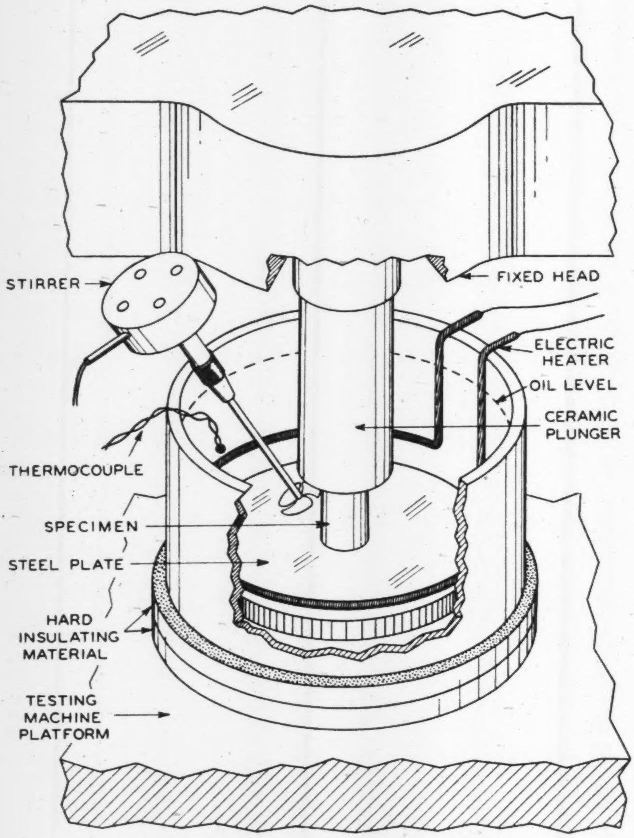
STIRRER
FIXED HEAD
ELECTRIC HEATER
OIL LEVEL
CERAMIC PLUNGER
THERMOCOUPLE
SPECIMEN
STEEL PLATE
HARD INSULATING MATERIAL
TESTING MACHINE PLATFORM

FIG. 1.—Sketch of the Compression Apparatus.

mercial cable sheath. The compositions are listed in Table I.

APPARATUS AND METHOD

The test consists in compressing cylinders of various materials at constant rates and temperatures and automatically recording the loads and corresponding deformations. The cylinders were cast to the finished diameter of 0.92 in. and microtomed to a length of 1.50 in. The mold used was a thin-walled nickel tube which rested on a cold aluminum base. The tube and melt were heated to 400 C prior to casting. This technique resulted in smooth sound castings having columnar grains starting from the base. While other casting methods produced radial grains, test results on such ingots differed but slightly in compression properties from those recorded in this paper. However, the relative effects of composition changes are similar in the two types of castings. The results here recorded are based entirely on castings made in the tube.

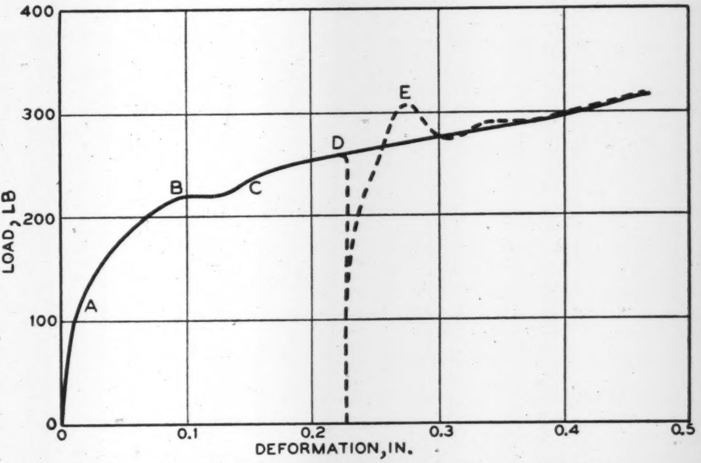

FIG. 2.—Compression Curve for Corroding Lead *C* at 220 C.
The effect of a 10 sec load-release is shown by the dotted curve.

The compression apparatus shown in Fig. 1 was used in an Emery-Tatnall testing machine equipped with a recording microformer deflectometer. Prior to each test, the samples were held in a thermostatically-controlled oil bath for 10 min. The bath rested on heat-insulating material, and the upper compression member was made of ceramic to reduce heat loss. While various compression rates were studied, the results here reported were obtained using a rate of 0.1 in. per min. Two or more samples were tested for each condition, and the agreement between runs was generally excellent; in many instances the curves coincided.

RESULTS

Typical curves drawn by the equipment are shown in Fig. 2, obtained on samples of corroding lead C at 220 C. The curve is approximately linear from the origin to point *A*. This is not to be construed as an elastic range but rather represents a region where the creep rate of the material is much lower than the rate of deformation. Beyond this, there is generally a curved section to some point *B*. During this portion of the test, work hardening probably takes place. On further deformation, the purer grades of lead (and some of the alloys at higher temperatures) show an inflection which may be attributed to recovery, polygonization, and perhaps recrystallization. A region of approximate linearity may then extend over the upper portion of the curves.

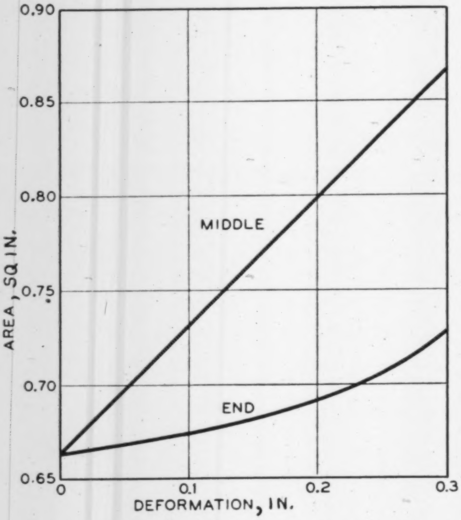

FIG. 3.—Area Variation at Indicated Sample Locations of Corroding Lead *C* at 220 C.

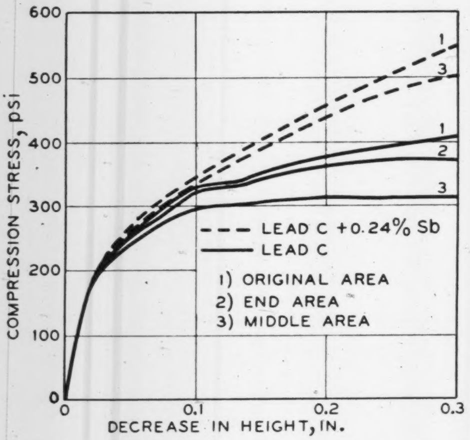

FIG. 4.—Compression Stresses of the End and Middle Areas.

The broken curve, starting at point *D*, shows the results obtained on some of the materials when the load was released for 10 sec and then reapplied. The form of the reloading curve closely resembles the tension test curves reported by Schumacher[4] on lead that had been cold worked and allowed to recrystallize at room temperature. In this latter instance, it was definitely observed that recrystallization commenced at a point corresponding to *E*. It is interesting to note that upon further deformation the load-deflection curve resumes a course quite similar to that obtained in an uninterrupted test.

It is recognized that parallel plate plastometry does not lend itself to mathematical treatment of the stresses involved.[5,6] After the early stages of deformation, there is a tendency for the samples to become barrel shaped. This is partly due to the friction between the ends of the specimens and the platens which restricts lateral motion at the contact surfaces during test. Figure 3 shows areas measured at the ends and middle of cylinders that were removed from test after different amounts of deformation. On the basis of the changing diameters, the compressive stresses at the ends and middle were calculated for samples of corroding lead C and of this material with 0.24 per cent antimony. These results are plotted on Fig. 4. Up to 0.1 in. deflection, the effect of barrelling is seen to be minor. At the higher deflections, it appears that deformation takes place in the midsection at constant stress for the pure lead whereas some work hardening may have occurred in the lead-antimony alloy. This conversion to stress values was not made for all of the materials tested,

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[4] E. E. Schumacher, "Metallurgy Behind the Decimal Point," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 188, p. 1097 (1950).

[5] A. Nadi, "Theory of Flow and Fracture of Solids," McGraw-Hill Book Co., New York, N. Y. (1950).

[6] C. E. Pearson, "The Extrusion of Metals," John Wiley, New York, N. Y. (1944).

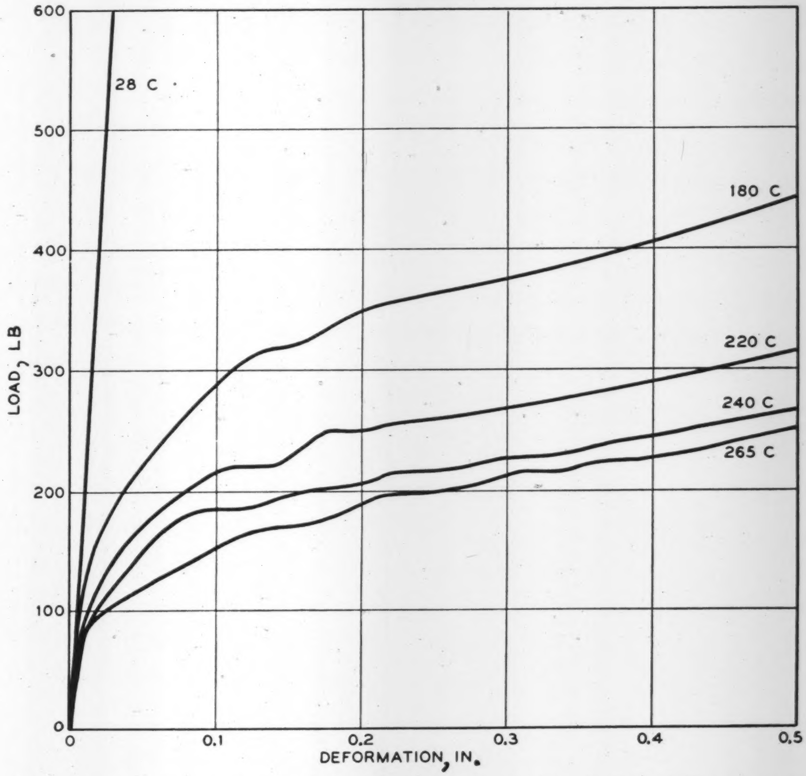

FIG. 5.—Load-Deformation Curves for Corroding Lead *A* at Indicated Temperatures.

since the differences in deformation behavior are apparent on the original curves.

Major differences between materials become evident after as little as 0.05 in. deformation, at which point the barrelling effect is nearly negligible. The loads required to produce comparable small deformation of the various materials may thus be considered as reliable indications of their relative plasticity. The slopes and the inflections of the individual curves beyond this point are probably sensitive indications of the changes taking place within the materials during continued deformation. This will be discussed more fully when considering the effects of alloying ingredients.

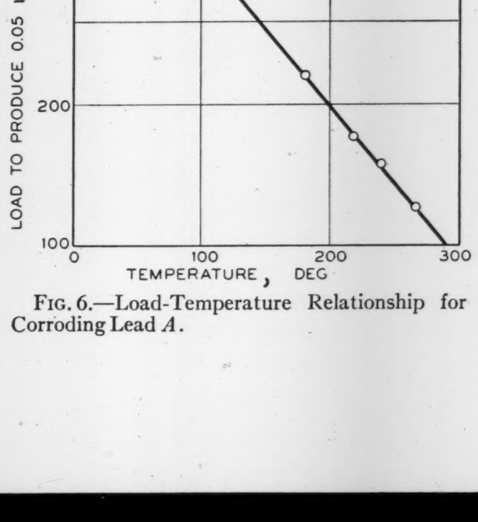

FIG. 6.—Load-Temperature Relationship for Corroding Lead *A*.

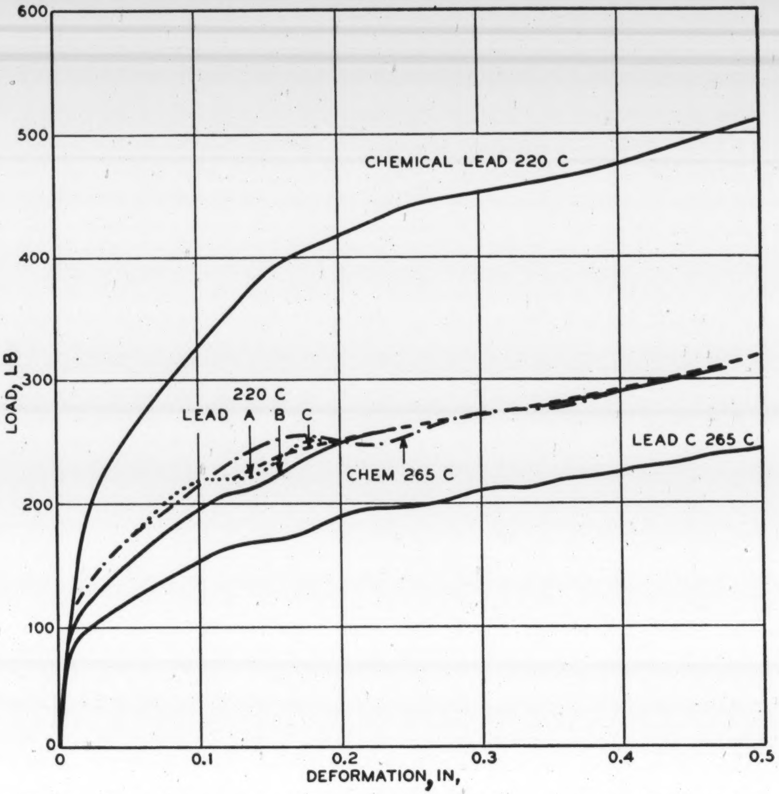

FIG. 7.—Load-Deformation Curves for Indicated Grades of Lead at 220 and 265 C.

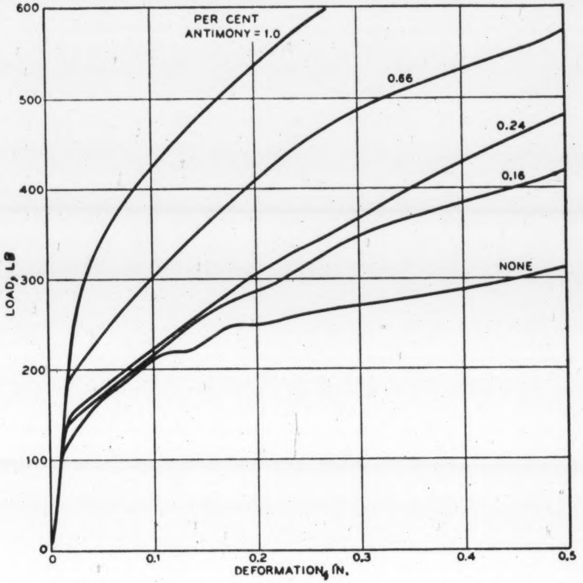

FIG. 8.—The Effect of Antimony on Corroding Lead *A* at 220 C.

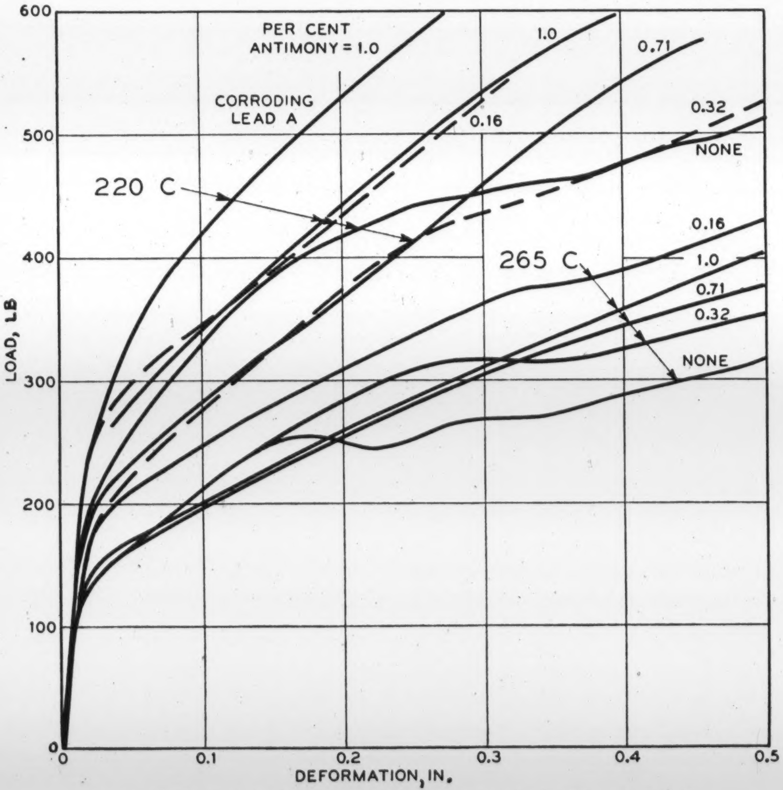

FIG. 9.—Effect of Antimony on Chemical Lead at 220 and 265 C.

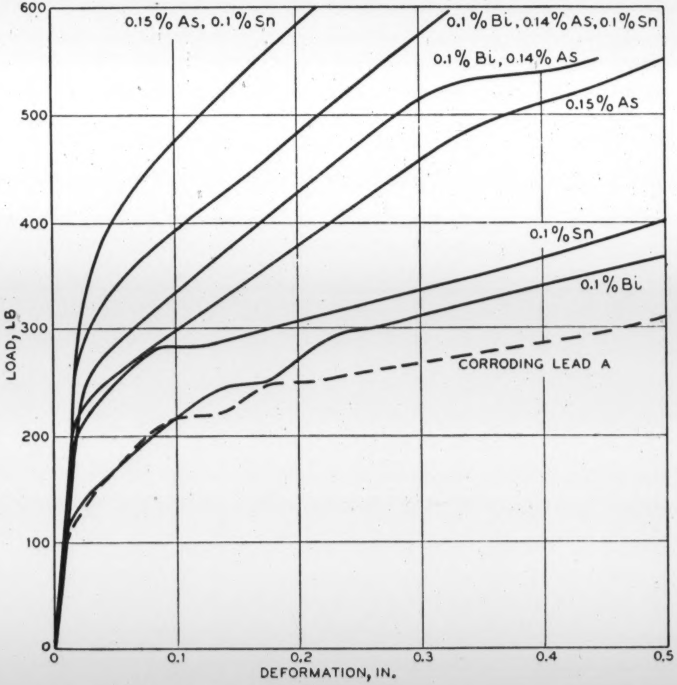

FIG. 10.—Load-Deformation Curves for Corroding Lead *A* Containing Other Elements.

Before discussing the behavior of the various materials, it will be of some interest to consider the effect of temperature. Figure 5 shows some results obtained on high-purity lead. As would be expected, the loads to produce given deformations are lower at the higher temperatures. This relationship is shown in Fig. 6, where the logarithm of the load, P, required to produce 0.05 in. deformation is plotted against temperature, T. The corresponding equation, $P = Ae^{-BT}$ is the same as the relationship reported by Pearson[6] for the variation of extrusion pressure with temperature for lead, aluminum, and other metals. A and B are constants depending on the material and e is the base of the natural logarithm. The correlation indicates that the present test method should yield information of value in predicting the extrusion characteristics of lead alloys.

Comparison of the load-deformation characteristics of commercial grades of lead at two temperatures is shown in Fig. 7. The three high-purity leads behave quite similarly at 220 C. The data indicate that chemical lead requires considerably more force for given deformations than lead *A*, *B*, or *C*. The chemical lead has an appreciable amount of copper disbursed in it as a second phase which might retard deformation; it is known to retard recrystallization. However, increasing the temperature from 220 to 265 C produced much greater lowering of the curve for chemical lead than it did for the purer lead. At 265 C, the chemical lead showed recovery indications similar to those for pure lead at 220 C except that greater deformation was required with the chemical lead before recovery was evident. Although no great change in solubility of copper in lead is expected between 220 and 265 C, it is known that at the higher temperature the primary copper needles tend to coalesce and become rounded, in which state they may be less effective in retarding recrystallization.

The effect of antimony on corroding lead A at 220 C is shown in Fig. 8. Even small amounts of antimony appear to retard recovery, and larger amounts appear to eliminate it completely. All of the antimony would normally be in solid solution at the test temperature. The effect of antimony on chemical lead at two temperatures is shown in Fig. 9. In this case, the effects are somewhat different than with pure lead. The anomalous behavior of the 0.16 per cent antimony alloy is of particular interest. It is known that copper and antimony form an intermetallic compound, the presence of which may have considerable influence on the high-temperature plasticity of chemical lead even though this amount of antimony has no unusual effect on the room temperature tensile strength characteristics of chemical lead. The curve for 1 per cent antimony in corroding lead has been included in Fig. 9 for comparison. Commercial experience indicates that 1 per cent antimony alloys made from chemical lead extrude more readily than those made from high-purity lead.

Considerable interest has been evident in recent years on the effect of arsenic in lead. The results of added arsenic and other elements on high-purity lead are shown in Fig. 10. Bismuth has but slight effect. The effect of tin is most apparent in the early stages of deformation. The curve continues the initial upward trend for a longer period than in the case of pure lead, indicating that the lead-tin may work harden to a greater extent. However, the inflection occurs after an amount of deformation comparable to that of pure lead. Arsenic causes a much longer initial straight portion and retards indications of re-

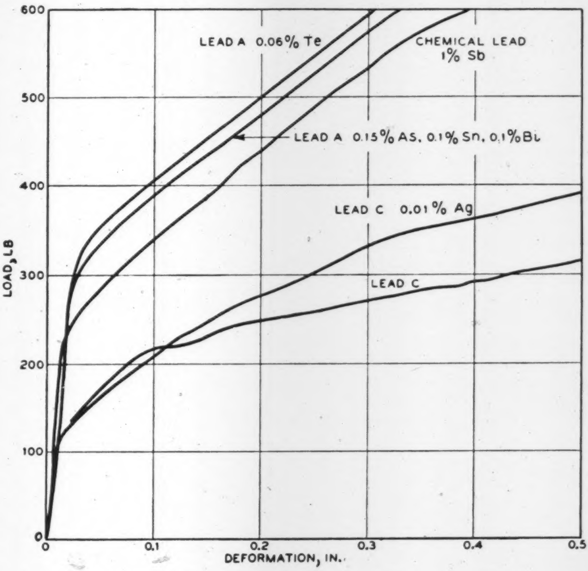

FIG. 11.—Load-Deformation Curves for Some Cable-Sheath Alloys at 220 C.

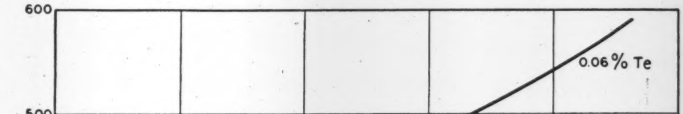

FIG. 12.—Load-Deformation Curves for Some Cable-Sheath Alloys at 265 C.

covery until higher amounts of deformation. Tin plus arsenic cause an appreciable rise in the load curve while the addition of bismuth to the combination causes a reduction in load.

Comparison of some of the more important cable sheath alloys is given in Figs. 11 and 12 at 220 and 265 C respectively. The silver causes only a minor change in deformation properties and is known to extrude in a manner quite similar to that of pure lead. The other materials are grouped together quite closely at 220 C, while at 265 C the lead-tellurium alloy appears much less plastic than the other materials.

DISCUSSION

MR. JOHN F. ECKEL[1] (*presented in written form*).—This paper is a real contribution to an important subject in the manufacture of cables. I have been interested for some time in the plasticity of lead as affected by alloying elements but more specifically in the manner in which extrusion pressure is affected. It is my belief that the compression test described by the authors, a similar compression test, or a small laboratory extrusion apparatus might serve well to determine rather accurately solid solubilities in systems where the solubility limits are uncertain but known to be very low.

Some time ago, we prepared a series of nine lead alloys containing arsenic up to 0.17 per cent and extruded each one in a small laboratory press of the inverted type. Although pressure, extrusion rate and temperature were about the same in each case, they were not controlled as closely as desirable for significant data. We did find very interesting results, however, when the extrusion pressure was plotted as a function of the arsenic content. At an extrusion temperature of 250 C, the pressure increased very rapidly to a value of about 8000 psi at 0.02 per cent arsenic. There was a sharp break in the curve at 0.02 per cent arsenic, with practically no change in extrusion pressure at higher arsenic contents. This break in the pressure-composition curve occurred at a point corresponding closely to the limit of solubility at 250 C as given by Bauer and Tonn.[2]

These results were interpreted to mean that most of the increase in extrusion pressure resulted from solution hardening and that (at least in this case) a secondary phase had little effect. It would be interesting to know whether the authors have any compression data that would serve to indicate solubility limits in the alloys with which they have worked.

MR. G. M. BOUTON (*author*).—I should like to thank Mr. Eckel for his discussion and the interesting points he brought up in connection with our paper. The data he presented indicated that there is a good possibility of determining phase relationships by measuring the changes in plasticity at the higher temperatures, either by running a series of alloys at one temperature or by running an alloy that has a change of solubility at a series of temperatures.

We have done nothing along this line, although some of the data that we have indicates that there may be phase changes taking place; for example, the effect of a small amount of antimony on copper-bearing leads shown in Fig. 9.

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[1] Research Laboratory, General Electric Co., Schenectady, N. Y.

[2] O. Bauer and W. Tonn, "Die Arsen-Bleilegierungen," *Zeitschrift fur Metallkunde*, Vol. 27, p. 183 (1935).

THE INFLUENCE OF COLD WORK AND HEAT TREATMENT ON THE ENGINEERING PROPERTIES OF BERYLLIUM COPPER WIRE*

By J. T. Richards,[1] R. K. Levan,[1] and E. M. Smith[1]

Synopsis

Electrical conductivity and tension tests were performed on beryllium copper wire to determine the influence of cold work, solution treating and precipitation hardening. Grain size, preferred orientation, straightening, overstraining in tension, and stress relieving were found to have a marked effect upon tensile strength, proportional limit, ductility and tension modulus. The method of plotting stress-strain data has also been considered.

Beryllium copper wire has found increased application during the past few years for springs, wire forms and miscellaneous items on account of its strength, conductive qualities and corrosion resistance. It is generally furnished in one of several heat-treatable tempers to be formed or coiled, then subsequently precipitation hardened to the desired combination of final properties. Although there is also available pretempered beryllium copper wire, this paper is concerned only with the heat-treatable form.

Beryllium copper wire has been commercially supplied for twenty years, yet little has been written regarding its engineering properties. Several authors (**1**, **2**)[2] have treated the subject in a general way, while others (**3**, **4**) have called attention to certain processing characteristics. In perhaps the first paper devoted to beryllium copper wire, Bassett (**5**) determined the influence of beryllium content and cold work on the tensile strength, although the precipitation hardening features of the alloy were not known at the time. Two recent articles have listed typical design properties for commercial conditions and tempers (**6**, **7**).

The present paper is the first report of a continuing investigation. It is concerned chiefly with the influence of processing and fabricating variables on electrical conductivity and tensile properties. The effects of cold drawing and precipitation hardening determined from several preliminary tests have been briefly summarized elsewhere (**8**).

Since beryllium copper wire is generally furnished solution treated or in one of several cold-drawn tempers, the producing mill is generally responsible for the cold-drawing and solution-treating operations. The fabricator forms or coils the unhardened wire, then precipitation hardens it as required. To establish design and specification limits, some knowledge of the influence of thermal treatment and cold work is necessary. In addition, the results of straightening and overstraining are considered, since these operations are similar in many respects to some of the more common fabricating procedures.

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* Presented at the Fifty-fourth Annual Meeting of the Society, June 18–22, 1951.

[1] Development Engineer, Junior Metallurgist, and Metallurgist, respectively, The Beryllium Corp., Reading, Pa.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 790.

It should be pointed out that property data and heat-treating time and temperature relationships obtained for beryllium copper strip do not necessarily apply for wire. The greater reductions of area usually given wire during processing mean higher final strength and lower ductility compared to equivalent strip tempers. This is apparent in checking the present results against similar data for strip (9).

MATERIALS

The standard 1.90 per cent beryllium copper alloy meeting the requirements of ASTM Specification B 197[3] was studied in this investigation. Although a number of lots were tested, the material, for convenience, can be divided into lots N and P.

TABLE I.—CHEMICAL COMPOSITION.

Element	Composition, per cent
Beryllium	1.81 to 1.86
Cobalt	0.23 to 0.27
Iron	0.08 to 0.10
Silicon	0.06 to 0.09
Aluminum	0.01 to 0.07
Nickel	0.01 to 0.03
Tin	0.01
Lead	0.000
Zinc	0.00
Copper	Balance

Lot N includes wire in various conditions, ranging from 0.202 down to 0.020 in. diameter and having little or no preferred orientation. Lot P, on the other hand, had a marked preferred orientation and was obtained by drawing 84 per cent and solution treating. This particular lot was then further cold drawn up to 50 per cent reduction.

Chemical analyses are listed in Table I.

TEST METHODS

The tensile strength was determined in accordance with the Standard Methods of Tension Testing of Metallic Materials (E 8 – 46),[4] using specimens of full cross-sectional area. Tension tests were conducted on a 5000-lb Olsen testing machine. The proportional limit and various yield strength values were taken from stress-strain curves plotted from strain values obtained with a Baldwin indicating wire extensometer. The dial, graduated in 0.001 in. divisions, measures directly the relative displacement of the gage blocks over a 10-in. gage length. Since readings can be estimated to $\frac{1}{10}$ of a dial division, the sensitivity of strain values is of the order of 0.00001 in. per in.

In plotting a series of preliminary tests, it was found difficult to draw a straight line through more than four or five test values, especially where the tensile strength exceeded 100,000 psi. When the data were plotted on a relatively open scale, test points were found to form an arc.

Curve *a* in Fig. 1 shows a typical stress-strain curve plotted to 190,000 psi for solution-treated, straightened and precipitation-hardened wire. The proportional limit may be defined[5] as the greatest stress which a material is capable of developing without deviation from the law of proportionality of stress to strain (Hooke's law). According to this definition, the proportional limit for this curve would be 80,000 psi based on a tension modulus of 25,800,000 psi.

To determine the usefulness of this proportional limit value, the stress-permanent set relation for this specimen is given in curve *b* of Fig. 1. The elastic limit, which is the greatest stress that a material is capable of developing without a permanent deformation remaining upon complete release of the stress,[5] is approximately 160,000 psi. Since, by definition[5] the tension modulus is the ratio, within the elastic limit of the material, of stress to strain, the portion of the stress-strain curve below the elastic limit can conceivably have a modulus and proportional limit value for each tangent or straight line that can be fitted to the arc at various stress levels.

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[3] Tentative Specification for Beryllium Copper Alloy Wire (B 197 – 50 T), 1950 Supplement to Book of ASTM Standards, Part 2, p. 106.

[4] 1949 Book of ASTM Standards, Part 2, p. 993.

[5] Standard Definitions of Terms Relating to Methods of Testing (E 6 – 36), 1949 Book of ASTM Standards, Part 2, p. 986.

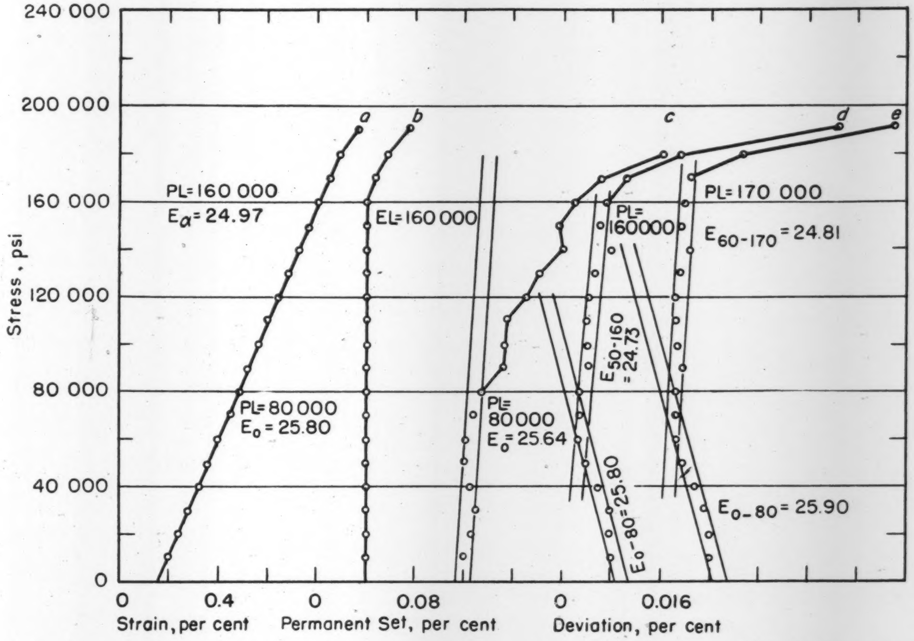

Fig. 1.—Stress-Strain Behavior of Beryllium Copper Wire, Solution Treated, Straightened and Precipitation Hardened 2 hr at 600 F (Lot P, 0.080 in. diameter).

(*a*) Nominal stress-strain curve.
(*b*) Uncorrected stress-set curve.
(*c*) Uncorrected stress-deviation curve (assumed E = 25,800,000 psi).
(*d*) Uncorrected stress-deviation curve (assumed E = 25,000,000 psi).
(*e*) Stress-deviation curve corrected for true stress and true strain (assumed E = 25,000,000 psi and μ = 0.35).

For all curves: E_0 = initial modulus, E_a = average modulus, PL = proportional limit, EL = elastic limit, and numerical subscripts = applicable stress range in 1000 psi. All tension modulus values given in 1,000,000 psi.

In an attempt to clarify this anomolous condition, a previously described (**10** to **17**) deviation method was employed. The values plotted are the difference between observed values at a given stress and those calculated from an assumed modulus. If the actual and assumed modulus are identical, points fall on a straight and vertical line; however, variations between the true and assumed moduli are indicated by a sloping line.

Curve *c* of Fig. 1 shows the stress-strain data of curve *a* plotted in this manner. If the proportional limit is the stress at which a straight line drawn between two consecutive points cuts the right-hand line of the scatter band, a value of 80,000 psi is obtained. Since the scatter band has a width equal to twice the least count of the extensometer, the proportional limit is based on an offset of 0.001 per cent from the true modulus line. Although similar proportional limit values result from the above two methods, the true modulus of 25,640,000 is somewhat lower than the assumed modulus of 25,800,000 psi taken from the original stress-strain curve.

In reconsidering the stress-strain curve *a* of Fig. 1, it was observed that a chord could be drawn to the arc, so that there fell on the chord the initial points and the last few values prior to the point where a definite yield or marked change of curvature could be noted. Since the slope of the chord is equal to the tangent modulus at the point of maximum depth of arc, it can be considered as the average modulus, E_a, for the elastic portion of the curve.[6] The proportional limit of 160,000 psi, which is established by the average modulus, agrees with the elastic limit fixed by the stress-set curve (curve *b*).

Curve *d* of Fig. 1 plots stress-deviation data based on an assumed modulus equivalent to the average modulus of curve *a*. The resulting points fall into two scatter bands having different slopes, while the proportional limit remains at 160,000 psi.

Anderson (18) has indicated that curvature observed in the elastic region of the nominal stress-strain diagram can frequently be eliminated by correcting for true stress and true strain. Since the true stress - true strain curve lies above and to the left of the usual curve, it is evident that what may appear to be deviation or yielding is, in reality, caused by failure to allow for the decrease in cross-sectional area and the increase in gage length with load. Anderson has devised the following simplified expression to obtain a single correction factor, which when added to observed strain values, gives a fairly accurate approximation of true stress - true strain conditions

$$\phi \cong -(1/2 + 2\mu)\epsilon_0^2 \ldots\ldots\ldots (1)$$

where:

ϕ = the strain correction,

μ = Poisson's ratio, and

ϵ_0 = the observed elongation on the original gage length.

It is apparent from the corrected stress-deviation curve *e* of Fig. 1 that the elastic region is still substantially curved; however, the proportional limit has been raised to 170,000 psi. In the case of all stress-deviation plots, strain values show a wave-like fluctuation within the band, similar to that observed by Smith (14).

Unless otherwise noted, the average modulus, E_a, has been employed in evaluating stress-strain data. In spot checking results, good agreement has been found to exist between the more precise method outlined in Fig. 1 and the average modulus procedure. Compared to the initial modulus, E_0, the average modulus E_a provides a more realistic approach for determining design stresses and constants.

Since the value of the proportional limit is dependent upon the sensitivity of the test method, it is qualified herein as the stress at 0.002 per cent offset from the average modulus.

Preliminary tests indicated that modulus determinations tended to be erratic for coiled wire, particularly in the soft or solution-treated condition. The low results obtained on first loading were probably due to the pulling out of small bends or kinks (10). If the proportional limit is exceeded slightly, subsequent tests give higher and more consistent values. In all tests, therefore, the specimen was first loaded slightly above the proportional limit, the load released and then immediately reloaded to fracture. Although coil set decreased modulus, it did not affect proportional limit or yield strength determinations. Consequently, these latter figures were taken from the initial stress-strain curve, while the highest average modulus obtained from the two curves is considered the most typical. Plotted or listed values are the average of two to ten tests.

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[6] The average modulus could also be considered as the secant modulus to the curve at the proportional limit.

DISCUSSION OF DATA

Since this paper is concerned primarily with the influence of processing and fabricating variables on the characteristics of beryllium copper wire, many of the property variations noted result from extreme conditions and are not found in normally processed material. For example, preferred orientation, which is produced by solution treating after heavy cold reductions, has a marked effect upon many of the results. This influence can be readily observed by comparing lots N and P.

Solution Treating:

To determine the effect of solution-treating time and temperature, a series of tests were conducted on 0.080-in. diameter wire which had previously been reduced 84 per cent in area by cold drawing. Specimens were heated in an uncontrolled atmosphere for 7, 15, 30, 45, and 60 min at 1400, 1470, and 1540 F,

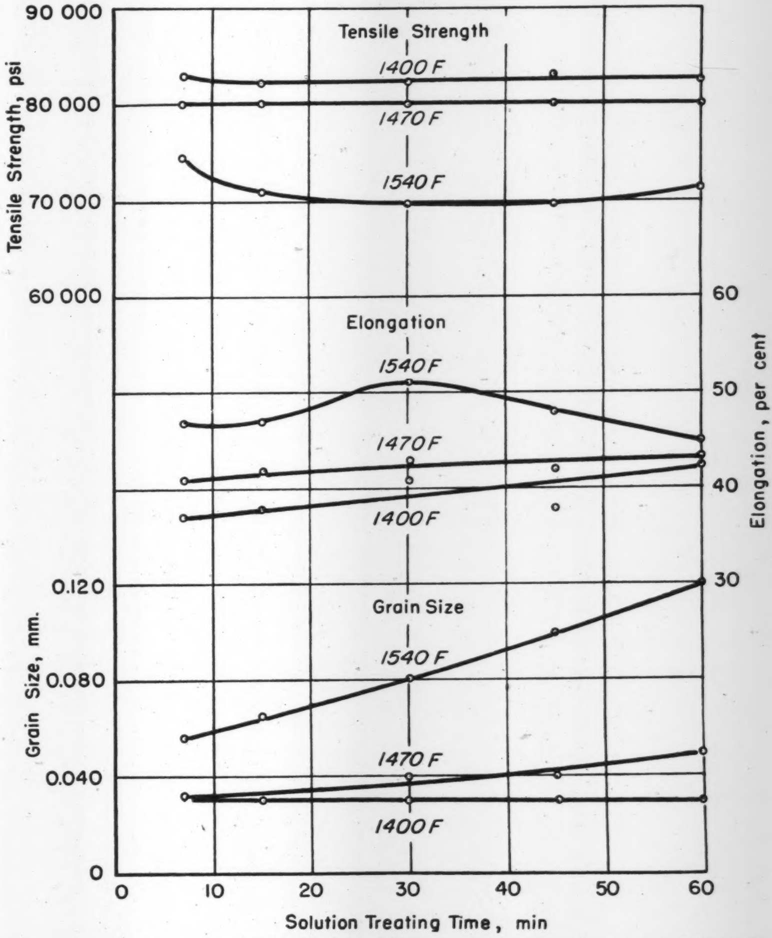

FIG. 2.—The Influence of Solution-Treating Time and Temperature on the Tensile Strength, Elongation and Grain Size of Beryllium Copper Wire (Lot P, 0.080 in. diameter).

then immediately quenched in cool water.

Figure 2, which shows the influence of solution-treating time on tensile strength, elongation, and grain size, indicates that property changes are slow to moderate at 1400 and 1470 F but fairly rapid at 1540 F. Considerable scatter is encountered with the large grain sizes obtained from the 1540 F treatment, so that the results are not particularly reliable. In a manner similar to beryllium copper strip (9), wire frequently shows reduced elongation at large grain sizes through premature failure.

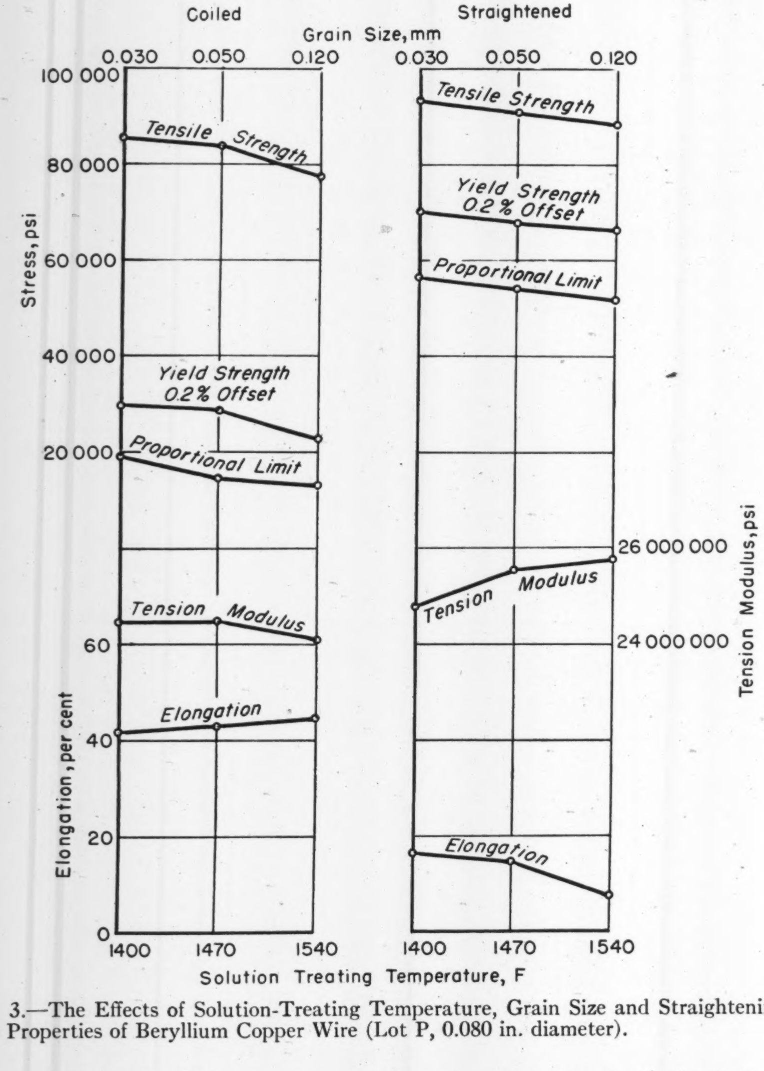

FIG. 3.—The Effects of Solution-Treating Temperature, Grain Size and Straightening on Some Tensile Properties of Beryllium Copper Wire (Lot P, 0.080 in. diameter).

In Fig. 3 the effects of solution-treating temperature and straightening on tensile and yield strength, proportional limit, tension modulus, and elongation are plotted. As might be expected, cold work resulting from machine straightening with rotating adjustable dies increases the strength with a marked decrease in elongation. No explanation is offered regarding the unexpected increase in modulus caused by straightening; however, this trend is believed to be real in view of the numerous tests conducted. The unusually high tensile strengths (approximately 15,000 to 20,000 psi above normal) are probably due to preferred orientation, as will be discussed later.

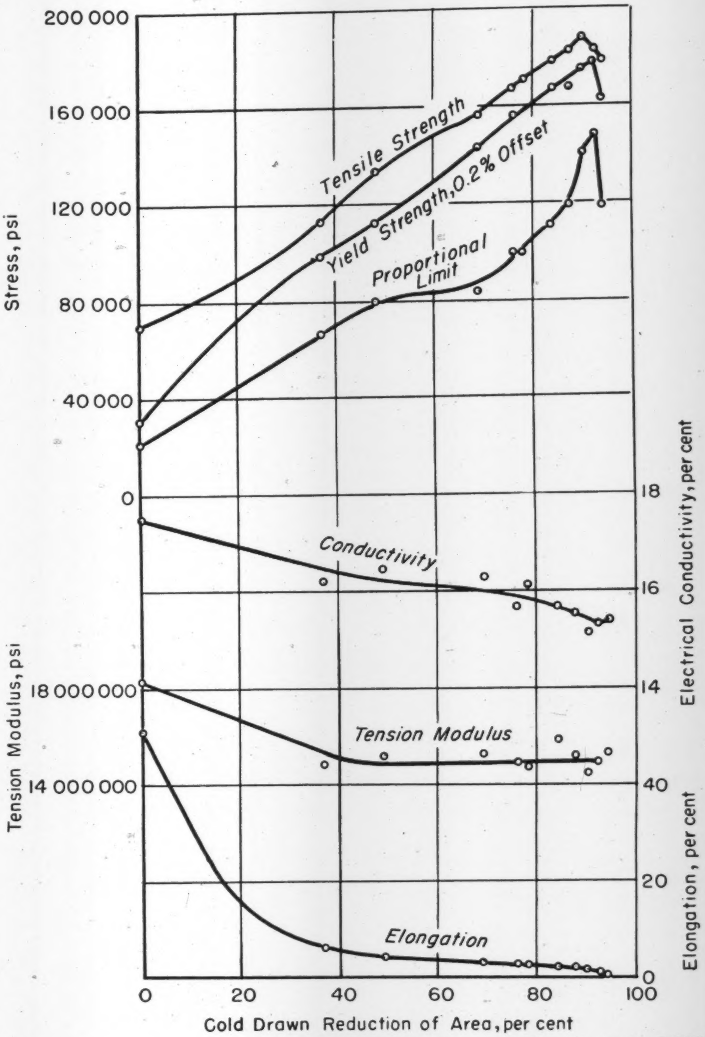

FIG. 4.—Cold-Drawing Characteristics of Beryllium Copper Wire (Lot N, 0.202 to 0.028 in. diameter).

On the basis of these tests, 1470 F is perhaps the most useful solution-treating temperature. Time at temperature should range from 30 to 60 min, depending upon wire size, with such additional furnace time as may be necessary to allow the charge to reach temperature.

Cold Drawing:

As shown by Fig. 4, cold drawing increases tensile strength, yield strength, and proportional limit but decreases electrical conductivity, tension modulus and elongation. It is of interest to note that proportional limit and tensile and yield strength values peak between 90 and 92.5 per cent reduction of area, then drop off rapidly with further cold work. In practice, final cold reductions rarely exceed 50 per cent; however, the data presented for higher tempers may be applicable to wire that is severely cold formed during fabrication. The sharp increase in proportional limit between 80 and 92.5 per cent reduction of area was unexpected, since in the case of copper and most copper alloys the proportional limit increases at a relatively slow rate beyond 50 per cent reduction (19).

TABLE II.—WORK HARDENING RATES OF SEVERAL COPPER ALLOYS COMPARED.

Wire Material	Tensile Strength at Stated Reduction of Area, psi		Difference in Tensile Strength, psi
	0 per cent	84 per cent	
Beryllium copper, 2 per cent Beryllium	69 000	177 000	108 000
Phosphor bronze, 5 per cent tin	50 000	140 000	90 000
Copper silicon, 3 per cent silicon	60 000	145 000	85 000
Brass, 70-30	51 000	130 000	79 000
Copper, electrolytic tough pitch	35 000	66 000	31 000

The decrease in electrical conductivity with cold drawing, 10.3 per cent in the case of 84 per cent reduction agrees with the 11 per cent value obtained by Crampton, Burghoff, and Stacy (20) for a beryllium copper alloy containing 1.53 per cent beryllium.

Cold drawing causes a decrease in tension modulus of approximately 18 per cent. From an initial value in the solution-treated condition of 18,300,000 psi, the modulus drops gradually to around 15,000,000 psi at 40 per cent reduction of area, and then is relatively constant through 94 per cent reduction. In view of the scatter shown in Fig. 4, the course of the modulus curve is, at best, an approximation. A similar decrease with cold work has been confirmed for many metals and alloys by other investigators (15, 16, 17, 21).

Not unlike other materials, beryllium copper shows a rapid initial drop in elongation with cold drawing, followed by a more gradual decrease. For solution-treated wire the elongation generally ranges between 40 and 60 per cent; the higher values applying to wire of larger diameter. In a recent investigation of the mechanical properties of beryllium copper wire, Pogodin and coworkers (22) found a consistent drop in elongation with wire size from 5.0 per cent for 0.170 in. to 1.5 per cent for 0.032 in. diameter cold-drawn material.

Table II compares the work hardening rates of several copper alloys on the basis of tensile strength difference between 0 and 84 per cent cold-drawn reduction of area.

Precipitation Hardening:

Since beryllium copper is a precipitation-hardening alloy, its characteristics can be altered by means of a suitable, low-temperature treatment. From Fig. 5 it is apparent that the property improvements resulting from heat treating are in addition to those gained through cold work, although the increase in strength is greatest for solution-treated material.

The influence of precipitation-hardening times and temperatures on wire cold drawn 50 per cent and straightened are shown in Fig. 6. All of these curves are based on heating in a recirculating air furnace and should be considered only as typical. Some allowance should be made for different heating rates, faster in the case of a salt bath or slower for a muffle furnace.

Tensile strength, proportional limit, and yield strength increase with time to a maximum and then fall off with overaging. By increasing the precipitation-hardening temperature, the time required to reach peak strength is reduced.

It is of interest that the proportional limit, which may play an important part in the selection of design stresses, is more stable and consequently does not overage as rapidly as yield strength or tensile strength values.

The electrical conductivity, as shown by Fig. 6, increases with time and temperature, while the tension modulus increases rapidly at first and then

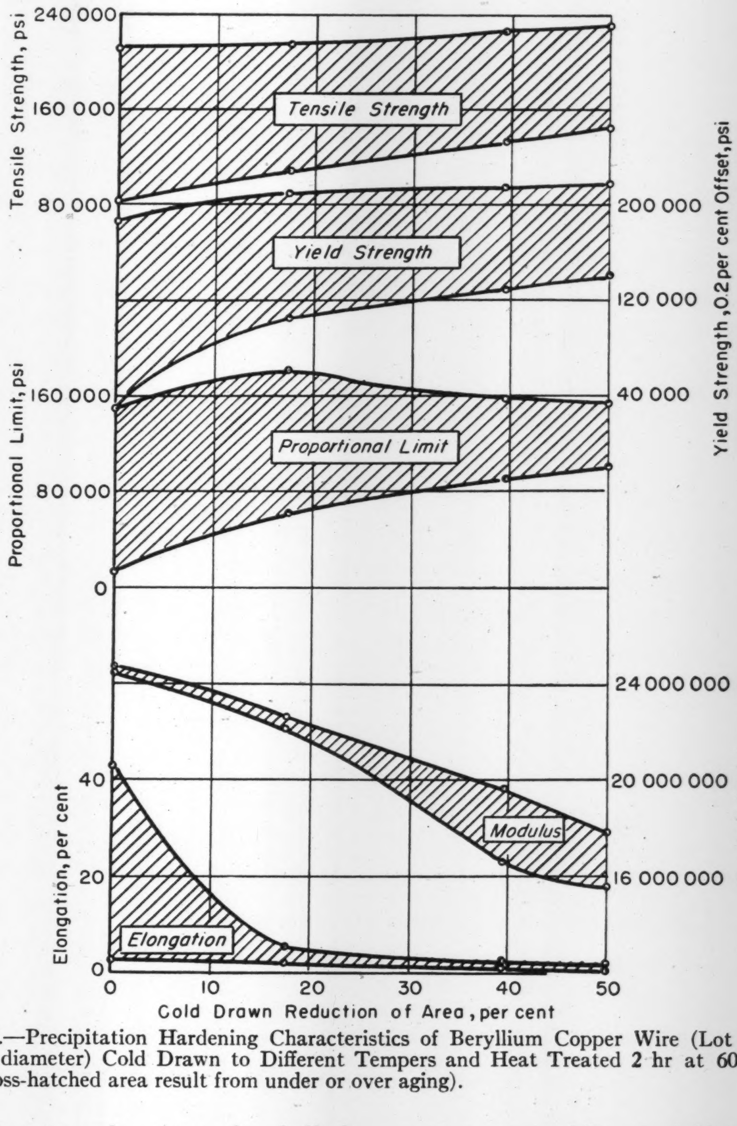

Fig. 5.—Precipitation Hardening Characteristics of Beryllium Copper Wire (Lot P, 0.080 to 0.057 in. diameter) Cold Drawn to Different Tempers and Heat Treated 2 hr at 600 F (Values within cross-hatched area result from under or over aging).

becomes relatively constant. Although other investigators report an increase in modulus with heat treatment, there is no general agreement as to the amount. Masing and Haase (**23**) claim that modulus may be improved, depending upon precipitation-hardening time and temperature, up to 30 per cent. On the other hand, Guillet (**24**) found an increase of only 6 per cent, while Smith and Van Wagner (**16**) noted a rise of 9 to 10 per cent in precise tests on rod. In the

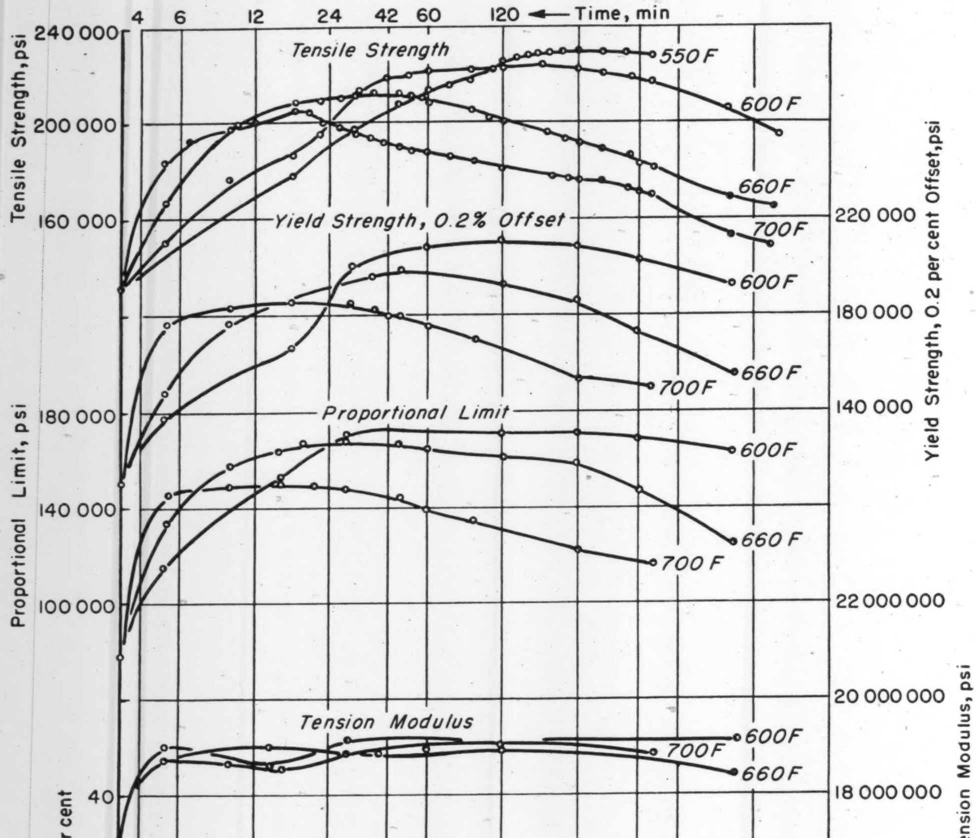

FIG. 6.—The Influence of Precipitation-Hardening Time and Temperature on Beryllium Copper Wire (Lots N and P 0.057 in. diameter) Cold Drawn 50 per cent.

present investigation, the improvement averaged 12 per cent for lot P (preferred orientation) and 11 per cent in lot N (no apparent orientation). For an aluminum-magnesium alloy (12 per cent magnesium), an increase of 4 per cent due to heat treating has been noted (12).

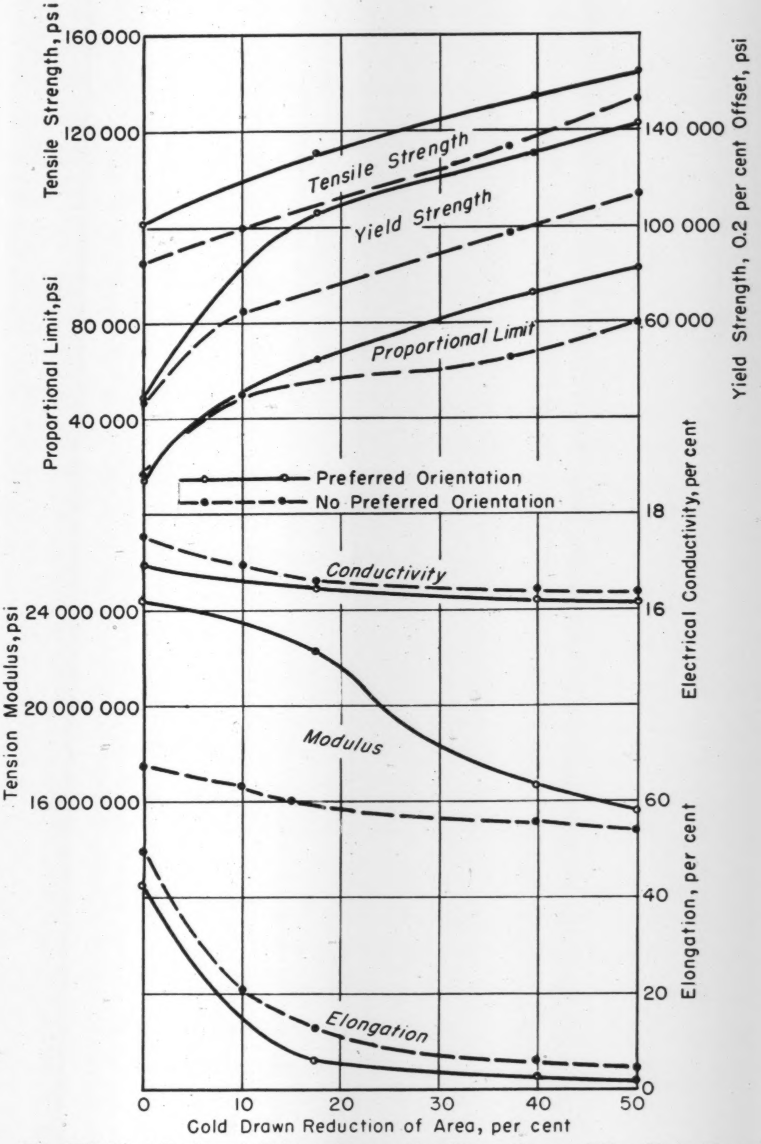

Fig. 7.—The Effect of Preferred Orientation on Beryllium Copper Wire (Lots N and P, 0.080 to 0.050 in. diameter).

Preferred Orientation:

The unusually high tensile strength and tension modulus of the 0.080-in. diameter solution-treated material (lot P) suggest the presence of preferred orientation. It is apparent from these properties that the cold-drawn texture was not completely removed by recrystallization during solution-treating. The influence of preferred orientation is indicated in Fig. 7 by comparing lots N and P. Lot N was cold drawn 44 per cent and lot P 84 per cent prior to similar solution treatments at 0.080-in. diameter.

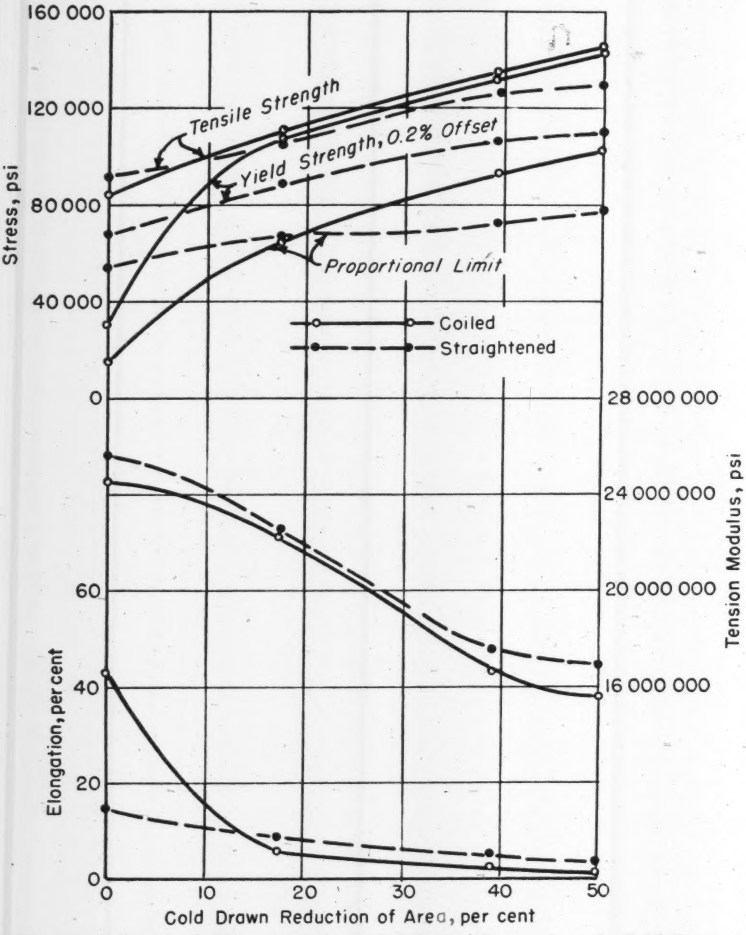

FIG. 8.—The Straightening Characteristics of Solution-Treated and Cold-Drawn Beryllium Copper Wire (Lot P, 0.080 to 0.057 in. diameter).

Perhaps the most unusual result of preferred orientation is its effect on tension modulus. The values obtained for solution-treated wire ranged from 24,000,000 to 26,000,000 psi and are believed to be the highest yet reported for beryllium copper in any form. As indicated by Fig. 5, precipitation hardening further increases the modulus. Figure 7 shows that cold drawing rapidly reduced the modulus, so that at 50 per cent reduction it approaches usual values. In considering other metals and alloys, wide variations in modulus depending upon crystallographic orientation have been observed in single crystals (25); however, these differences have not been as pronounced in polycrystalline forms.

These tests indicate that preferred

orientation can be produced or prevented in beryllium copper by controlling the amount of cold work. Insufficient data are available, however, to determine whether preferred orientation can be held to special limits to meet certain design requirements.

With increasing temper or cold-drawn reduction, the strengthening influence of straightening is progressively decreased, so that from approximately 8 to 20 per cent reduction there is little difference between coiled and straightened wire. As the temper is further increased, the tensile properties of coiled wire continue to rise rapidly, while those of the straightened wire increase at a lower rate. As shown in Fig. 8, the tensile strength, yield strength, and proportional limit of coiled wire drawn 50 per cent are substantially higher than those of the straightened material. At the same reduction, however, the elongation of straightened wire is approximately twice that of coiled wire. For all tempers considered, the

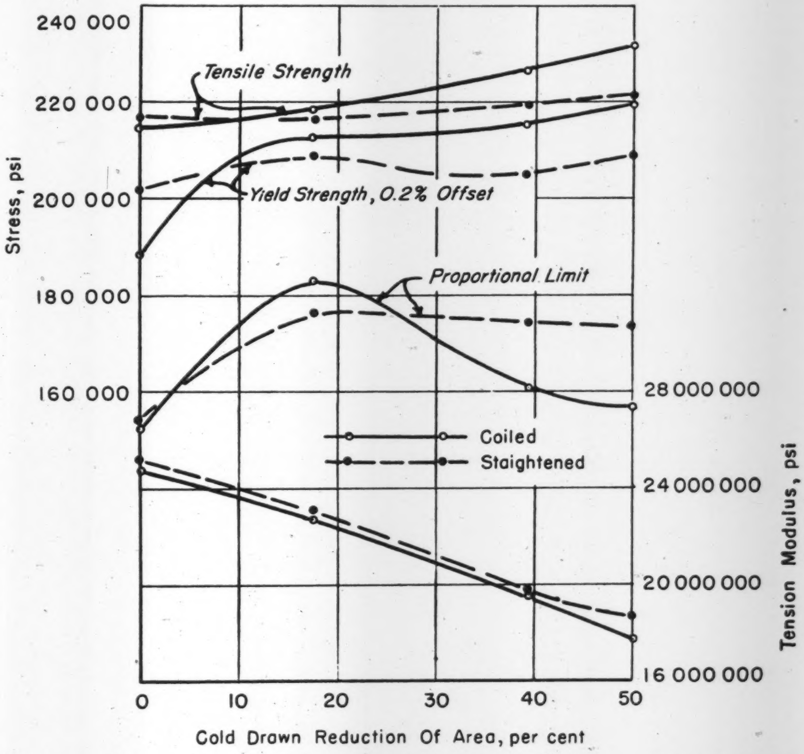

Fig. 9.—Some Effects of Precipitation Hardening for 2 hr at 600 F on Coiled and Straightened Beryllium Copper Wire (Lot P, 0.080 to 0.057 in. diameter) in Various Tempers.

Straightening:

As previously indicated in Fig. 3, machine straightening with rotating adjustable dies has a significant effect upon the properties of solution-treated wire. With the possible exception of tension modulus, the changes are in the same direction as those resulting from cold drawing. There is a slight increase in tensile strength accompanied by a more marked increase in yield strength and proportional limit.

tension modulus of straightened wire is higher than that of coiled wire.

These results are somewhat similar to those noted by Pungel (26) in machine-straightened cold-drawn steel wire. Pungel found that straightening decreased the tensile strength but increased the yield strength and elongation. In obtaining comparable data, Domes (27) also determined the magnitude of internal stresses and found that straightening reduced these stresses to a fraction of their original value. Consequently, it appears that property variations between coiled and straightened wire can be explained on the basis of varying combinations of strain hardening and stress relief. In the solution-treated condition, the influence of strain hardening is at a maximum and probably decreases rapidly with increasing temper. On the other hand, the effect of stress relief is at a minimum for solution-treated wire but increases with temper and reaches a relatively high value for severely cold-drawn stock.

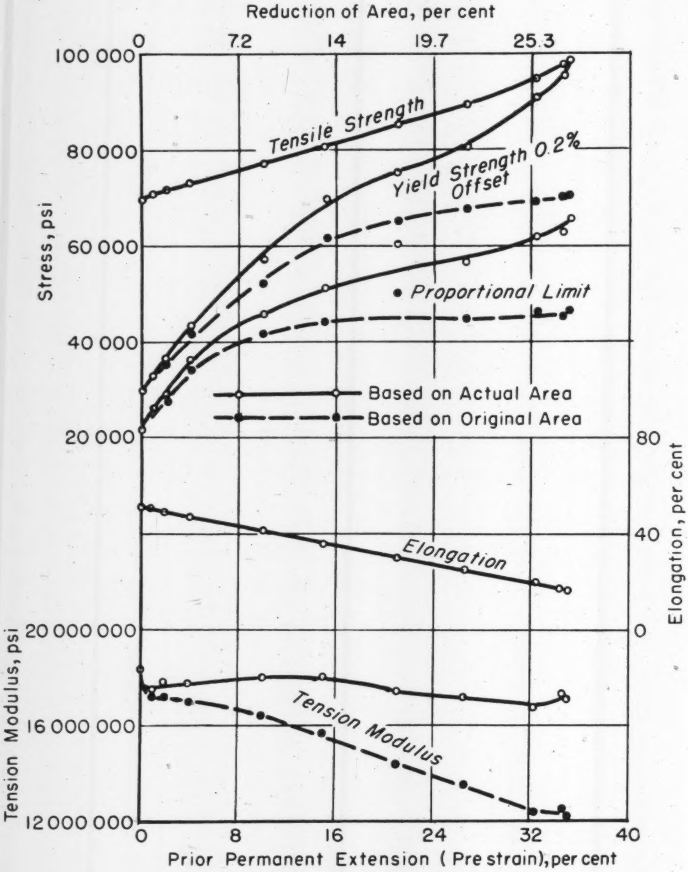

FIG. 10.—The Influence of Increasing Tensile Deformation or Overstrain on Some Tensile Properties of Solution-Treated Beryllium Copper Wire (Lot N, 0.202 in. diameter).

Figure 9 indicates the influence of precipitation hardening upon the properties of coiled and straightened wire. The results parallel, at a considerably higher stress level, those obtained prior to heat treating. A possible explanation for the irregular behavior of the proportional limit is its extreme sensitivity to internal stress, so that stress relief gained through straightening and precipitation hardening may outweigh the strain hardening factor at heavily cold-drawn tempers.

TABLE III.—THE EFFECT OF TENSILE OVERSTRAIN AND STRESS RELIEF ON TENSILE PROPERTIES OF BERYLLIUM COPPER WIRE.

Original Condition	Overstraining Stress, psi	Relieving Treatment		Tensile Strength, psi	Yield Strength, 0.2 per cent Offset, psi	Proportional Limit, 0.002 per cent Offset, psi	Tension Modulus, psi
		Time, hr	Temperature, deg Fahr				
1. Solution treated and straightened	None	None	None	91 500	64 900	52 900	25 600 000
	84 700	None	None	95 600	88 800	76 200	24 440 000
2. Drawn 50 per cent and straightened	None	None	None	130 100	110 100	77 200	17 000 000
	119 000	None	None	126 300	119 600	80 700	16 530 000
3. Drawn 84 per cent	None	None	None	177 800	172 000	120 000	15 780 000
	None	1	200	180 900	178 700	131 000	15 690 000
	None	1	300	183 200	183 000	151 000	16 330 000
4. Solution treated, straightened and precipitation hardened 2 hr at 600 F	None	None	None	206 800	200 000	150 900	25 200 000
	198 000	None	None	226 700	204 700	198 300	24 460 000
	198 000	2	400	212 000	206 700	203 200	24 780 000
5. Solution treated, straightened, precipitation hardened 2 hr at 600 F, prestressed to 198 000 psi and relieved 2 hr at 400 F	207 500	None	None	212 700	211 800	207 400	24 670 000
	211 700	None	None	216 300	215 900	211 700	24 770 000
6. Drawn 50 per cent, straightened and precipitation hardened 2 hr at 600 F	None	None	None	205 100	203 700	163 100	18 780 000
	200 000	None	None	212 800	210 000	167 800	18 750 000
	200 000	2	400	212 100	209 600	183 000	18 820 000

Tensile Overstrain and Stress Relief:

To provide a means of predicting results of certain fabricating operations, the influence of tensile overstrain on tensile properties was studied. Specimens in the solution-treated condition were loaded to produce given extensions, released, and then immediately retested. Overstrain for these tests ranged from zero to the maximum uniform extension occurring at the ultimate load. Elongation values were determined as the difference between the total extension of the original material at failure less prior permanent strain.

As shown in Fig. 10, plotted on the basis of both original area and actual area at the start of each run, proportional limit, tensile strength, and yield strength increase while elongation and modulus decrease with increasing tensile deformation.

In addition, other specimens were slightly overstrained during the course of stress-strain testing to insure straight wire for subsequent modulus determinations. Several specimens in various conditions were also stress-relieved prior to tension testing.

As indicated in Table III, both tensile overstrain and low-temperature stress-relieving can be depended upon to improve the proportional limit and yield strength, while maximum improvement results from a combination of the two treatments. Tension modulus is generally decreased by overstraining and increased by stress relieving. Through the selection of suitable overstraining and relieving treatments, it is possible to raise the proportional limit of a specimen above the original tensile strength (see items 4 and 5 of Table III). Similar results were achieved by Galibourg (**28**) with mild steel.

Several other features of overstraining merit attention. When the complete stress-strain cycle is plotted, the ascending and descending curves frequently do not coincide so that a hysteresis loop is formed. If the test specimen is stressed above its elastic limit, a permanent set will remain after the load is released. In beryllium copper wire, as in other materials (**13, 16, 17, 29**), hysteresis and permanent set decrease with cyclic stressing over the same load range.

According to Bauschinger (**30**), overstraining a material above the proportional limit but below the yield point raises its proportional limit; overstraining a material above the yield point (*a*) gives a decreased proportional limit upon immediate reloading, (*b*) increases its yield point to a stress equivalent to the previous load, and (*c*) slightly improves its tensile strength.

On the basis of this investigation, beryllium copper follows all of Bauschinger's principles with the exception that, in virtually every test, overstraining above either the proportional limit or the yield strength was found to increase the proportional limit.

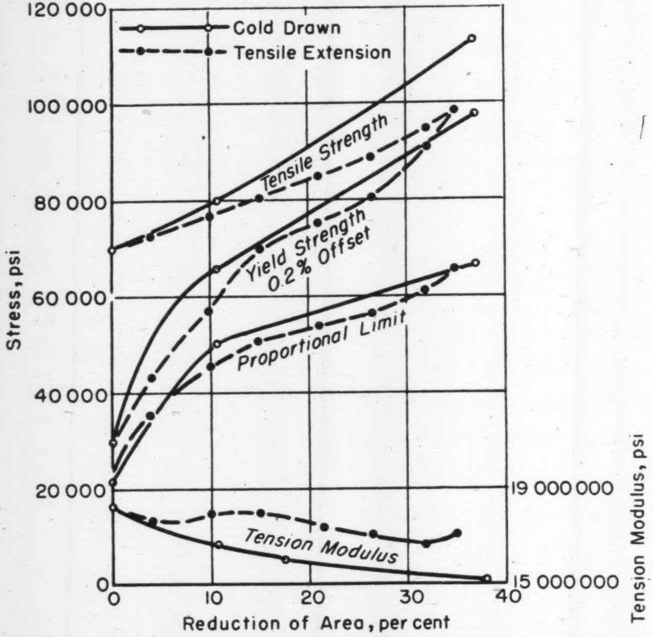

FIG. 11.—The Effects of Cold Drawing and Tensile Overstrain Compared for Beryllium Copper Wire (Lot N, 0.202 in. diameter).

Cold Drawing and Tensile Overstraining Compared:

Figure 11, which compares the results for tensile overstrain with cold drawing, shows good agreement for yield strength and proportional limit with wider variations apparent for tensile strength and modulus.

Jackson (31) and Voce (32) have recently shown that cold drawing is similar to tension and that strains due to drawing can be combined with those resulting from tension tests to form a continuous stress-strain curve. By so doing, a fundamental relationship for strain hardening can be established which is independent of the stress system producing the strain. To provide the generalized strain corresponding to various drawn reductions, Jackson has suggested the following equation

$$\bar{\epsilon}_r = \ln \frac{A_0}{A} \quad \ldots\ldots\ldots\ldots \quad (2)$$

where A_0 is the original area and A the final area.

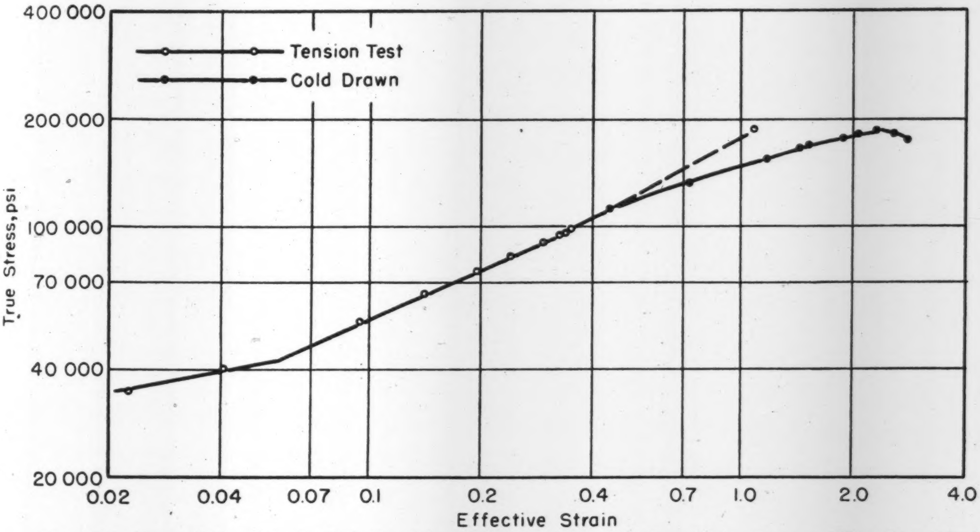

FIG. 12.—True Stress-Strain Relationship of Solution-Treated Beryllium Copper Wire (Lot N, 0.202 in. diameter) Subjected to Cold Drawing and Tensile Overstrain.

This relationship is shown in Fig. 12 for solution-treated beryllium copper wire that has been subjected to drawing and tension tests. The results give a linear plot on logarithmic coordinates between true stress and effective true strain for the tension test, while points representing various degrees of cold drawing fall on a prolongation of the tension curve. True stress can be determined by

$$\sigma = \frac{P}{A} \quad \ldots\ldots\ldots\ldots\ldots \quad (3)$$

where P is the load and A the instantaneous area, while effective true strain is

$$\delta = \ln \frac{l}{l_0} = \ln \frac{A_0}{A} \quad \ldots\ldots\ldots \quad (4)$$

where l and l_0 and A and A_0 are the instantaneous and original gage lengths and areas respectively.

Good agreement is found for the true stress - true strain relationship between beryllium copper strip (8) and wire. The break or sudden change in the tension curve is typical for copper-base alloys (33, 34), while the shape of the combined curve is similar to one previously constructed for copper (35).

The course of the broken line in Fig. 12, which connects the ultimate stress with fracture, is evidence of the dissimilarity existing between cold drawing and the necking region of the tension test.

Yield Strength:

Although yield strength values at 0.01 per cent offset and 0.5 per cent extension under load were determined from stress-strain curves, only the yield strength at 0.2 per cent offset has been reported.

Since the value at 0.01 per cent offset is close to the proportional limit (generally well within 10,000 psi), it can be readily estimated.

Though frequently employed for copper alloys, the yield strength at 0.5 per cent extension under load has limited value as in index of plastic damage (16). This is especially true for beryllium copper wire, even before precipitation hardening. As shown in Fig. 13, the proportional limit exceeds this yield value at reductions over 37 per cent, so that the entire specified extension can be produced elastically and the yield value is dependent solely upon the tension modulus.

CONCLUSIONS

1. Solution treating beryllium copper wire increases elongation but lowers tensile properties, including tensile strength, yield strength, and proportional limit. Low solution-treating temperatures give smaller grain sizes, higher tensile properties, and decreased elongation.

2. On cold drawing, tensile properties increase almost linearly up to 90 to 93 per cent reduction of area, then fall off with further reductions. Electrical conductivity, tension modulus, and elongation decrease with cold drawing.

3. Precipitation hardening increases tensile properties, conductivity, and tension modulus but decreases elongation. Although tensile properties reach a maximum with time and overage upon further heating, the individual properties do not necessarily peak at the same time. With increasing heat-treating temperatures, hardening times can be reduced. Electrical conductivity increased continuously throughout the test period.

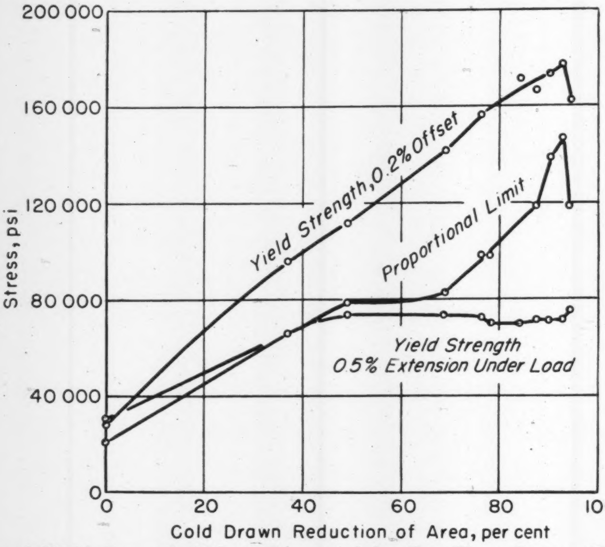

FIG. 13.—Several Yield Strength Values Compared for Beryllium Copper Wire (Lot N, 0.202 to 0.050 in. diameter).

4. Preferred orientation, which may result from excessive cold reductions, gives higher tensile properties and tension modulus with decreased conductivity and elongation.

5. Machine straightening increases tensile properties in the solution-treated material but decreases them in wire drawn over 15 to 20 per cent. The opposite is true for elongation, while tension modulus is increased slightly in all tempers. Similar trends were obtained for material precipitation hardened after straightening.

TABLE IV.—SUMMARY OF TYPICAL RESULTS FOR BERYLLIUM COPPER WIRE IN COIL FORM.
Lot N. Wire with marked preferred orientation (lot P) excluded.

	Solution-Treated		Quarter Hard		Half Hard		Three-Quarter Hard	
Heat treated 2 hr at 600 F	No	Yes	No	Yes	No	Yes	No	Yes
ASTM designation	A	AT	¼ H	¼ HT	½ H	½ HT	¾ H	¾ HT
Average cold drawn reduction, per cent	0	0	21	21	37	37	50	50
Tensile strength, psi	66 000	186 000	88 000	200 000	112 000	210 000	137 000	216 000
Yield strength, 0.2 per cent offset, psi	27 000	161 000	75 000	195 000	100 000	200 000	121 000	197 000
Proportional limit, 0.002 per cent offset, psi	15 000	126 000	49 000	155 000	71 000	160 000	84 000	159 000
Elongation in 2 in., per cent	50	5	14	3	5	2	3	1
Tension modulus, 1,000,000 psi	17.8	19.0	16.8	18.5	15.6	17.8	15.4	17.1
Electrical conductivity, per cent I.A.C.S.	17.5	24.0	16.8	23.0	16.4	22.6	16.3	22.3
Tensile strength (ASTM B 197 – 50 T) psi	58 000 to 78 000	160 000 to 190 000	90 000 to 115 000	175 000 to 205 000	110 000 to 135 000	185 000 to 215 000	130 000 to 155 000	190 000 to 220 000

6. Overstrain improves tensile properties with some reduction in elongation and modulus. Tensile overstrain and cold drawing produce similar results, although the mechanics of these operations vary.

7. Stress relieving heat-treatable or precipitation-hardened wire gives higher yield strength, proportional limit, elongation and tension modulus; however, the effect upon tensile strength varies. Optimum elasticity for a given condition results when stress relieving is combined with overstraining.

8. Yield strength methods have been considered and values obtained at either 0.01 or 0.2 per cent offset are perhaps the most useful. Yield strength at 0.5 per cent extension under load is of questionable value in the case of beryllium copper wire.

9. Since the elastic portion of the stress-strain relationship for beryllium copper is curved throughout, an average tension modulus is believed to be of greater practical value than the initial modulus.

10. Table IV lists typical properties for beryllium copper wire resulting from this study.

Acknowledgment:

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REFERENCES

(1) J. Delmonte, "Beryllium-Copper Wire Has Unique Characteristics," *Wire and Wire Products*, Vol. 11, pp. 326, 327, 355 (1936).

(2) "Age-Hardenable Beryllium-Copper," *Wire Industry*, Vol. 12, pp. 195–197 (1945).

(3) F. S. Stickney, "Making Spring Grade Beryllium Copper Wire," *Wire and Wire Products*, Vol. 19, pp. 168–172 (1944).

(4) M. J. Donachie, "Some Traits and Characteristics in the Working of Beryllium-Copper Wire," *Wire and Wire Products*, Vol. 20, pp. 719–721 (1945).

(5) W. H. Bassett, "Beryllium Copper Alloys," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 75a, pp. 218–232 (1927).

(6) J. T. Richards, "Beryllium Copper as a Spring Material," *Machinery* (New York), Vol. 55, April, 1949, pp. 169–174.

(7) J. T. Richards, "Beryllium Copper," *Materials and Methods*, Vol. 31, April, 1950, pp. 75–90.

(8) J. T. Richards, "A Review of Beryllium and Beryllium Alloys," *Journal of Metals*, Vol. 3, pp. 379–386 (1951).

(9) J. T. Richards and E. M. Smith, "The Forming Characteristics of Beryllium Copper Strip," *Proceedings*, Am. Soc. Testing Mats., Vol. 50, pp. 1085–1100 (1950).

(10) R. G. Batson and J. H. Hyde, "Mechanical Testing," Vol. 1, "Testing Materials of Construction," Chapman and Hall, Ltd., London (1922).

(11) Discussion by L. B. Tuckerman of paper by R. L. Templin, "The Determination and Significance of the Proportional Limit in the Testing of Metals," *Proceedings*, Am. Soc. Testing Mats., Vol. 29, Part II, pp. 538–546 (1929).

(12) R. L. Templin and D. A. Paul, "Modulus of Elasticity of Aluminum Alloys," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 93, pp. 480–486 (1931).

(13) D. J. McAdam, Jr. and R. W. Mebs, "Tensile Elastic Properties of 18-8 Chromium-Nickel Steel as Affected by Plastic Deformation," *Report No. 670*, National Advisory Committee for Aeronautics (1939).

(14) C. S. Smith, "Proportional Limit Tests on Copper Alloys," *Proceedings*, Am. Soc. Testing Mats., Vol. 40, pp. 864–872 (1940).

(15) D. J. McAdam, Jr. and R. W. Mebs, "Tensile Elastic Properties of Typical Stainless Steels and Nonferrous Metals as Affected by Plastic Deformation and by Heat Treatment," *Report No. 696*, National Advisory Committee for Aeronautics (1940).

(16) C. S. Smith and R. W. Van Wagner, "The Tensile Properties of Some Copper Alloys," *Proceedings*, Am. Soc. Testing Mats., Vol. 41, pp. 825–845 (1941).

(17) R. W. Mebs and D. J. McAdam, Jr., "Elastic Properties in Tension and Shear of High Strength Nonferrous Metals and Stainless Steel—Effect of Previous Deformation and Heat Treatment," *Technical Note No. 1100*, National Advisory Committee for Aeronautics (1947).

(18) Discussion by A. R. Anderson of paper by C. S. Smith, "Proportional Limit Tests on Copper Alloys," *Proceedings*, Am. Soc. Testing Mats., Vol. 40, pp. 877–881 (1940).

(19) R. A. Wilkins and E. S. Bunn, "Copper and Copper Base Alloys," McGraw-Hill Book Co., Inc., New York, N.Y. (1943).

(20) D. K. Crampton, H. L. Burghoff and J. T. Stacy, "Effect of Cold-work upon Electrical Conductivity of Copper Alloys," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 143, pp. 228–241 (1941).

(21) W. Koster and K. Rosenthal, "Changes in Elastic Modulus and Damping of Brass During Deformation and Recrystallization," *Zeitschrift für Metallkunde*, Vol. 30, pp. 345–348 (1938).

(22) S. A. Pogodin, N. K. Abrikosov and N. M. Elkhones, "Properties of Beryllium Bronzes with Lower Beryllium Content and with Additions of Other Components," *Izvestiya Sektora Fiziko-Khimichaskogo Analiza*, Institut Obshehei i Neorganicheskoi Khimii Akademii Nauk U.S.S.R., Vol. 16, No. 2, pp. 197–210 (1946).

(23) G. Masing and C. Haase, "The Changes in the Modulus of Elasticity During the Age-Hardening of Beryllium-Copper Alloys," *Wiss. Veroffentlich. Siemens-Konzern*, Vol. 8, No. 1, pp. 142–148 (1929); also "Beryllium," translation by R. Rimbach and A. J. Michel, Reinhold Publishing Corp., New York, pp. 171–179 (1932).

(24) L. Guillet, Jr., "Contributions to the Study of the Modulus of Elasticity of Metallic Alloys," *Revue de Metallurgie*, Vol. 36, pp. 497–521 (1939).

(25) M. F. Sayre, "Elastic Properties and Their Relationship to Strength and Strain

Hardening," Chapter from "Working of Metals," Am. Soc. Metals, Cleveland, pp. 932–942 (1937).

(26) W. Pungel, "Influence of Straightening and Subsequent Tempering of Steel Wire upon the Mechanical Properties," *Stahl und Eisen*, Vol. 62, pp. 854–856 (1942).

(27) V. Domes, "Influence of Treatment of As-drawn Wire upon Properties and Stress Conditions," *Stahl und Eisen*, Vol. 62, pp. 879–880 (1942).

(28) J. Galibourg, "Influence of the Amount of Elongation in Successive Loadings Followed by Aging on the Elastic Limit and Young's Modulus of a Mild Steel," *Comptes rendus*, Vol. 195, pp. 1022–1024 (1932).

(29) J. Muir, "On the Recovery of Iron from Overstrain," *Philosophical Transactions*, Vol. 193A, pp. 1–46 (1900).

(30) J. Bauschinger, "On the Change of the Elastic Limit and the Elastic Modulus of Various Metals," *Civilingenieur*, Vol. 27, pp. 289–347 (1881).

(31) L. R. Jackson, "Work Hardening and Rupture in Metals," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 171, pp. 622–638 (1947).

(32) E. Voce, "True Stress-Strain Curves," *Metal Treatment*, Vol. 15, pp. 53–66, 72 (1948).

(33) J. H. Hollomon, "Tensile Deformation," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 162, pp. 268–289 (1945).

(34) R. S. French and W. R. Hibbard, Jr., "Effect of Solute Elements on the Tensile Deformation of Copper," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 188, pp. 53–58 (1950).

(35) L. R. Jackson, A. M. Hall and A. D. Schwope, "The Comparative Properties of Several Types of Commercial Copper, As Cold Worked and As Recrystallized," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 175, pp. 296–305 (1947).

FATIGUE TESTS IN AXIAL COMPRESSION*

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SYNOPSIS

This paper presents the preliminary results of an investigation of the behavior of materials subjected to repeated compressive loads. Exploratory studies have been conducted on gray cast iron, 24S-T alluminum alloy, and steel. In general, compressive failures occurred only under high stresses. In structural steel (ASTM Specifications A 7), for example, failures were not observed at less than five million cycles until stresses in the neighborhood of 85,000 psi or higher were applied. These results, under nearly uniformly distributed axial compressive loads, are in distinct contradiction to results obtained with notched specimens or with repeated bending tests.

Most fatigue investigations have been conducted within a stress range which is either entirely tension or which varies from tension to compression. Relatively few tests have been made to determine the behavior of metals subjected to pulsating compressive loads alone. Moore, Lyon, and Inglis (**1**)[2] tested gray cast iron (static compressive strength of 111,000 psi) under repeated axial compression. The plot of their test results gave an S-log N curve similar to that obtained from tests conducted under repeated tensile stresses but which indicated substantially higher stresses. Their compressive endurance limit (zero to compression) was reported as 65,000 psi compared to the value of 15,500 psi for a cycle of zero to tension.

Nishihara and Kojima (**2**) tested an aluminum alloy while Nishihara and Sakurai (**3**, **4**) tested two types of cast iron and several medium- and high-carbon steels. Their results, given in Table I, indicate compressive endurance limits (based on 10 or 20 million cycles of stress) for all of the metals tested. In both the cast iron and aluminum specimens, the fatigue fractures occurred along diagonal planes of maximum shear and were similar to static compression fractures. Results have been reported also (**5**) on compressive fatigue failures in specimens of 17S-T (heat-treated duralumin) rod.

Very little information is available regarding the basic cause of fatigue failures. There are a number of hypotheses, some of which are plainly absurd. It is clear from the results of even the few tests available that the endurance limit is much higher under repeated compression than under repeated tension. Therefore any theory, such as the maximum shear stress theory or the maximum energy of distortion theory, which predicts equal strength under these two types of loading is plainly in error.

Because of the lack of information on

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[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 803.

the subject, this paper is primarily of an exploratory nature. The first part of the paper is concerned with the development of suitable specimens, equipment, and operating techniques which eliminate, as far as possible, many of the uncertainties existing in the state of stress of a compression fatigue specimen during the loading cycle. The specimens were fabricated with parallel ends and with smooth, polished surfaces in order to reduce the possible existence of stress raisers and to make the stress distribution across the specimen as uniform as possible. The apparatus was carefully designed and constructed to permit axial application of known loads with a minimum amount of bending in the specimen. By taking these precautions the results, while not directly applicable to specific structures, provide basic information on the compression fatigue strength of several materials.

TABLE I.—RESULTS OF PREVIOUS WORK.

Material	Compressive Endurance Limit, psi (10 000 000 cycles)	Static Tensile Strength, psi	Static Compressive Strength, psi	Yield Strength, psi
Cast iron, Moore, Lyon and Inglis **(1)**	65 000	31 600	111 000	....
Cast iron "A", Nishihara and Sakurai **(3)**	41 100	26 000	87 800	....
Cast iron "B", Nishihara and Sakurai **(3)**	58 700	32 600	89 300	....
Steel (0.41 per cent C), Nishihara and Sakurai **(4)**	100 000	100 700	....	55 200
Steel (0.44 per cent C), Nishihara and Sakurai **(4)**	85 100	88 000	....	56 700
Steel (0.65 per cent C), Nishihara and Sakurai **(4)**	103 000	105 000	....	55 700
Steel (0.90 per cent C), Nishihara and Sakurai **(4)**	116 300	....	....	....
Duralumin, Nishihara and Kojima **(2)**	37 100 (20 000 000 cycles)	56 000	72 000	31 900
Duralumin, Alcoa **(5)**	32 000 (20 000 000 cycles)	55 000	....	32 000

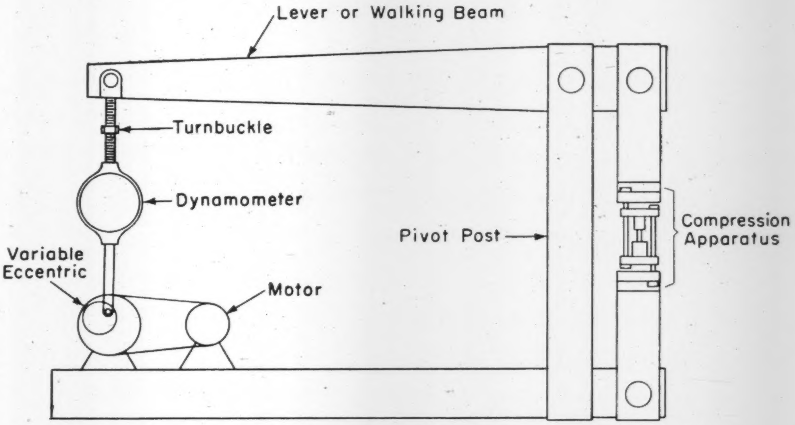


FIG. 1.—Diagrammatic Sketch of the Wilson Fatigue Testing Machine.

The second major part of this paper presents the test results obtained for specimens of gray cast iron, 24S-T aluminum alloy, mild steel, and other metals when subjected to repeated loads.

SPECIMENS AND TESTING PROCEDURE

Fatigue Testing Machine:

The testing machine used in this program is the W. M. Wilson type fatigue machine. It has a capacity of ±50,000 lb and operates at a speed of 290 cpm. Figure 1 is a diagrammatic sketch of the machine. The dynamometer on the left is used to measure the applied load. This load is controlled by the turnbuckle or adjusting screw and by the variable eccentric. The adjustment of the variable eccentric determines the range in load and the turnbuckle is used to position the maximum and minimum loads during a cycle. At the right of the figure is the pivot post and the compression apparatus.

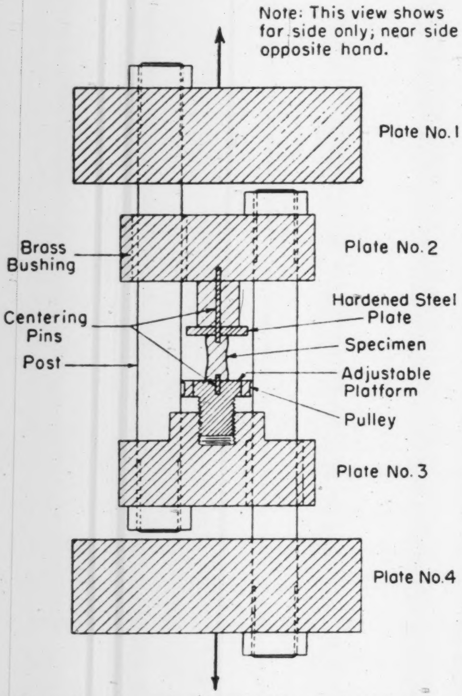


FIG. 2.—Cross-Sectional View of the Compression Apparatus Used in the Wilson Machine.

A cross-sectional view of the compression apparatus is shown in Fig. 2. The specimen rests on a hardened steel platform that can be adjusted in height by means of a screw thread. A weight and pulley arrangement applies a uniform torque to this platform and keeps it in constant contact with the specimen even if the specimen shortens during the test. The bearing surfaces, both above and below the specimen, have projecting steel pins which center the specimen and prevent it from moving laterally. Two diagonally opposite posts, fixed in plate No. 2, transmit the load from the lower pullhead of the machine to the upper end of the test specimen. The other two posts, fixed in plate No. 3, transmit the load to the lower end of the test specimen. This arrangement is quite stable and makes it possible to apply a compressive load to the specimen by the application of a tensile load to the compression apparatus.

Preparation of the Specimens:

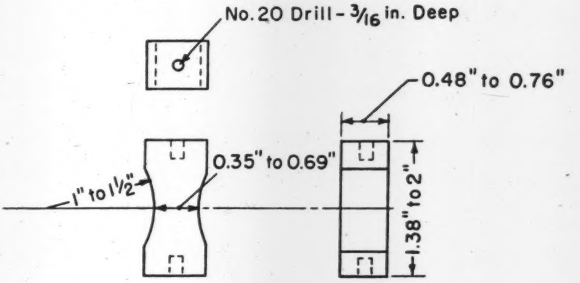
Details of the first specimens tested in this program are shown in Fig. 3(*a*). The materials tested with this type of specimen include gray cast iron and the other metals listed in Table II with the exception of killed structural steel. These specimens (Fig. 3(*a*)) had a rectangular cross-section which was slightly reduced on two sides. The other two sides of the specimen were machined flat so that the propagation of cracks could be observed.

This type of specimen was machined to final dimensions and then loaded in the fatigue machine. Since the maximum test stress of these specimens was greater than the yield strength of the individual materials, it was necessary to cold work the specimen by gradually increasing the load during the first few cycles. The specimen was then capable of resisting the desired high compressive stress without further permanent distortion.

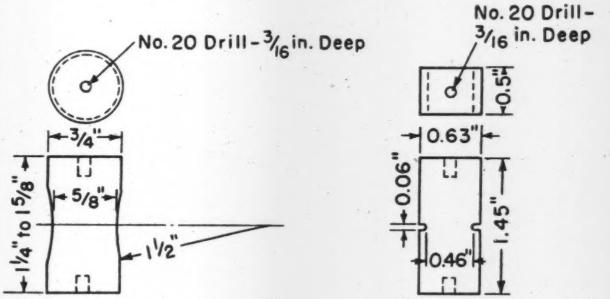
TABLE II.—RESULTS OF EXPLORATORY REPEATED COMPRESSION TESTS ON MILD STEEL AND OTHER METALS.

Materials	Static	Repeated Compression		
	Compressive Yield Strength, psi	Specimen	Maximum Test Stress, psi	Number of Cycles Applied
Killed structural steel	43 000	No. RK-6	89 000	2 147 600[a]
		No. RK-3	89 000	381 200[a]
		No. RK-4	84 000	5 360 000[b]
		No. RK-2	59 500	10 331 000[b]
		No. RK-1	53 000	12 409 100[b]
Annealed rail steel	44 800	No. B-1	130 000	1 000 000[b]
		No. B-3	110 000	2 005 000[b]
		No. B-5	95 000	2 521 300[b]
		No. B-2	71 000	1 076 400[b]
Rail steel as-rolled	72 500	No. 4-9	82 000	2 034 900[b]
Rimmed structural steel	39 000	No. RR-12	54 000	2 509 600[b]
Brass	44 000	No. RR-2	58 000	2 138 800[b]
Ductile cast iron	55 000	No. DC-1	67 000	2 272 800[b]
Chromium-molybdenum steel	125 000	No. Q-4	126 000	2 266 900[b]

[a] Number of cycles at which first crack appeared.
[b] No failure occurred.


No. 20 Drill - 3/16 in. Deep
0.48" to 0.76"
0.35" to 0.69"
1" to 1½"
1.38" to 2"

(a) Rectangular


No. 20 Drill - 3/16 in. Deep
3/4"
5/8"
1¼" to 1⅝"
1½"
No. 20 Drill - 3/16 in. Deep
0.5"
0.63"
0.06"
0.46"
1.45"

(b) Circular (c) Notched

FIG. 3.—Details of Specimens.

Similar specimens (Fig. 3(*b*)) were used for the tests on 24S-T aluminum and killed structural steel. Although they were about the same length as those of cast iron, these specimens had a circular cross-section. After the final machining had been completed, the surface of the specimen was polished to eliminate machining marks. This procedure reduced, as far as possible, the stress raising effect of the machining marks and made the appearance of fatigue cracks more evident. This specimen was considered superior to the one used for the tests of gray cast iron in that it provided a larger uniform surface upon which fatigue cracks could develop.

Because of the difficulty in applying a high stress to the cast iron specimens, a slightly different procedure was followed in preparing and testing the aluminum and the killed structural steel specimens. The stock material was turned down to a $\frac{3}{4}$-in. diameter cylinder, the ends were machined flat and parallel, and centering holes were drilled. These cylinders were then preloaded statically in a 120,000-lb hydraulic testing machine using a compression device which insured an axial load. During the pre-loading the diameter of the specimen was measured with a micrometer so that the nominal true stress could be computed. After the load necessary to produce an average stress of approximately 1000 psi more than the intended maximum fatigue test stress had been reached, this load was maintained until the deformations no longer increased. The specimen was then machined to its final dimensions, and the curved surface of the specimen was polished to remove the machining marks.

Although the test specimens for the general program were designed to reduce stress concentrations, one annealed rail steel specimen which had an 0.08-in. deep notch on each edge (Fig. 3(*c*)) was tested. This test was run primarily to determine whether the notch would reduce the fatigue strength in repeated compression as much as it would in repeated tension tests.

Testing Techniques:

It was difficult to maintain the desired stress on the gray cast iron specimens because of their initial plastic action. As the load was applied, the specimen shortened and the adjustable platform under the specimen rose, keeping the specimen in contact with the upper bearing plate. As the strain in the material increased, the area of the specimen became larger and the range of strain to which the specimen was to be subjected required an increasingly larger range of applied load. The progressive shortening of the specimen was evident only during the first few cycles of load application. After this initial shortening, the length of the specimen and the magnitude of the applied load became relatively constant.

In order to eliminate the difficulties and uncertainties in stress which were introduced by the progressive deformation of the specimen, the previously described pre-loading technique was developed for the tests of aluminum and killed structural steel specimens. Since the test stress was slightly below that applied in the pre-load, the specimens behaved elastically when loaded in the fatigue machine and their dimensions remained unchanged during the entire test.

In all of these fatigue tests, the minimum stress of the cycle was a compressive stress of about 1000 psi. The applied load was checked frequently and whenever the maximum stress dropped as much as 1000 psi below the desired stress the required load was reset. During the tests at high stresses the load had to be readjusted slightly about once a day.

Strain Distribution Within the Specimen:

Although great care was taken to insure an axial loading, the true stress distribution within the specimen was uncertain. In order to obtain a better understanding of the actual nature of the applied load, SR-4 electrical resistance strain gages were mounted at the reduced section of a typical aluminum specimen which had been preloaded and machined to final dimensions. Three Baldwin type AD-7 strain gages were mounted, at 120-deg intervals, parallel to the longitudinal axis of the specimen. The gages were wired so that individual strain readings could be taken as the load was applied.

Static and fatigue loads were applied to the calibration specimen while it was in the fatigue machine and the corresponding strain readings were taken on each of the gages. Although the limitations of the gages prevented testing above approximately 50,000 psi, definite trends were evident and could be extrapolated to the higher stresses.

From the three individual strain readings taken at a given load, the maximum and minimum values of strain within the specimen were determined and the corresponding eccentricity of the applied load was computed. A plot of the variation of the eccentricity with mean stress, as shown in Fig. 4, indicates that, although considerable eccentricity exists in the specimen at the lower stresses, it reduces rapidly and is less than 0.03 in. at the higher stress levels. For all orientations of the specimen, this calibration indicated that the maximum compressive strain occurred in the same position relative to the testing machine. When one considers the size of the fatigue machine and compression rig as compared with the specimen, this value of eccentricity is about as small as machining tolerances will permit.

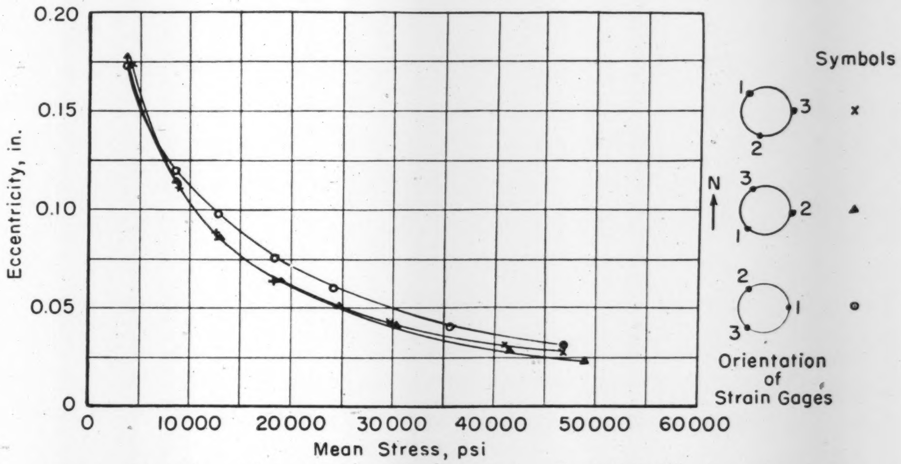


FIG. 4.—Variation of Eccentricity with Mean Stress of a 24S-T Aluminum Specimen.

At the very low stress levels, the individual strain readings indicated that a slight tension existed on one side of the specimen. However, after a mean stress of approximately 15,000 psi had been

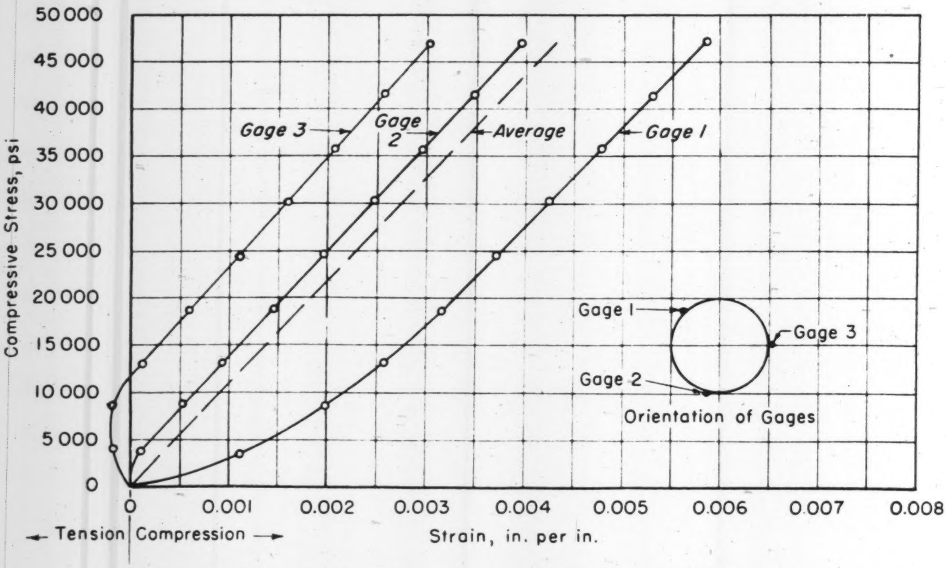


FIG. 5.—Stress-Strain Calibration of a 24S-T Aluminum Specimen.

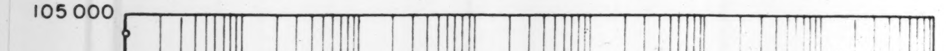
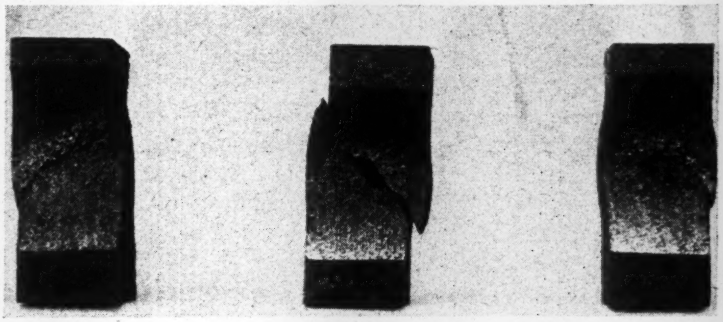
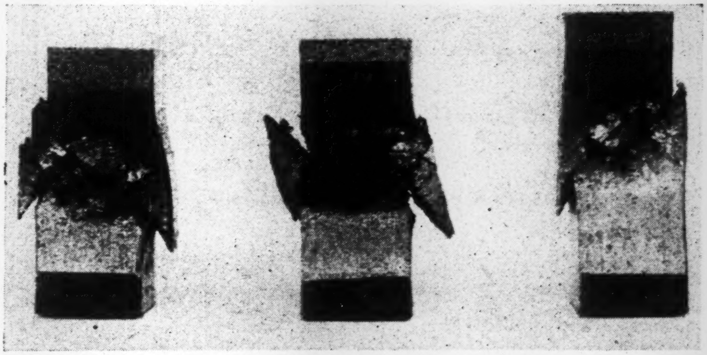


FIG. 6.—Results of Repeated Compression Tests of Gray Cast Iron.

attained, each gage indicated compressive strains which increased in a linear manner as additional load was applied to the specimen. This strain variation is shown in Fig. 5.



(*a*) Static Tests.



(*b*) Fatigue Tests.
Fig. 7.—Compression Fractures of Gray Cast Iron.

PRESENTATION AND DISCUSSION OF TEST RESULTS

Gray Cast Iron:

The results of the fatigue tests on gray cast iron (static compressive strength of 95,000 psi) are plotted to a semi-log scale in Fig. 6. The large amount of scatter is probably the result of the extremely heterogeneous structure of the material. A straight line has been drawn through the minimum test values giving a curve of minimum specimen life for the various applied stresses.

The gray cast iron specimens fractured under repeated compressive loading in the same general manner as the static specimens, that is, in a diagonal shear type failure. This is illustrated by the three static failures shown in Fig. 7(*a*), and the three typical repeated compression failures shown in Fig. 7(*b*). It is evident in these photographs that the specimens which failed in fatigue were, in general, more broken up than the ones which were tested statically. During the tests, the fatigue specimens bulged and appeared to be progressively breaking up inside before they finally crumbled into several pieces.

24S-T Aluminum:

The results of the tests on 24S-T aluminum (static compressive yield strength, 0.1 per cent offset, of 48,000 psi) are plotted to a semi-log scale in Fig. 8. Since the aluminum proved to be more resistant to crack propagation under repeated compressive loads than the gray cast iron, it was considered advisable not to test the specimens to fracture as was done for the cast iron specimens. In these tests the initial detection of a surface crack was taken as the criterion for failure. Under a stress of 83,000 psi, the initial crack in specimen No. 2 occurred after 170,400 cycles of stress. After 35,000 additional cycles of stress this crack was clearly visible as shown in Fig. 9.

The cracks which appeared at the surface of the specimen were readily visible to the unaided eye. Apparently when a crack formed, the edge served as a reflector for light and hence was seen easily. A commercial dye penetrant[3] was used to confirm the cracks and also to disclose the possible existence of minute cracks which were not yet visible.

In every aluminum specimen tested,

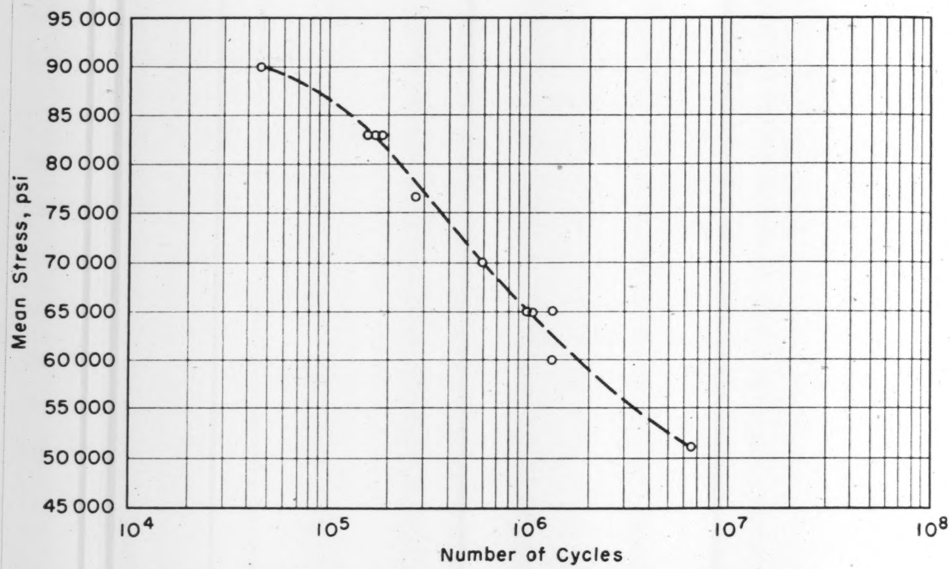


FIG. 8.—Results of Repeated Compression Tests of 24S-T Aluminum.

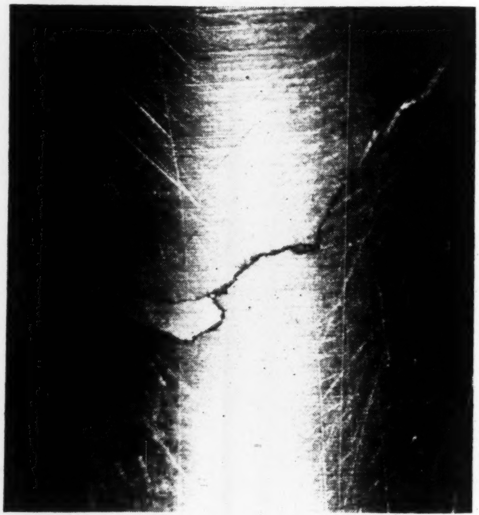


FIG. 9.—Aluminum Specimen No. 2, 35,000 Cycles After Appearance of First Crack. (×20.)

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[3] The penetrant used was Dy-Chek, a product of the Dy-Chek Co., Hawthorne, Calif.

the initial cracks appeared in the region where the previously described calibration tests had shown the maximum compressive strains to be present. However, when additional cycles of stress were applied to the specimens, these initial cracks spread and new ones developed around the entire circumference of the net section.

The ability of the specimens to sustain load remained unchanged even after the cracks had spread to advanced stages. The dimensions of the specimen likewise remained unchanged insofar as could be determined with a 0.001-in. micrometer.

Based on failure at 1,000,000 cycles, the fatigue strength of 24S-T aluminum is about 26,000 psi for a stress cycle in which the stress varies from tension to an equal compression and approximately 43,000 psi for a stress cycle in which the stress varies from zero to tension. At the same number of cycles the results of the compressive fatigue tests reported herein indicate a fatigue strength of about 65,000 psi.

Mild Steel and Other Metals:

The results of the preliminary tests on specimens of killed structural steel (Table II) indicate that at stresses below 84,000 psi compressive fatigue failures will not occur at less than 5,000,000 cycles. From the results of rotating-beam tests the fatigue strength of the killed steel was found to be approximately 42,000 psi for one million cycles of stress. No data are available for the fatigue strength of the material under a load varying from zero to tension; however, a value of about 55,000 psi would be expected for failure at 1,000,000 cycles.

Several tests were conducted on specimens of annealed rail steel. As indicated in Table II none of these specimens developed fatigue cracks even though one was subjected to 130,000 psi for 1,000,000 cycles.

Because of the relatively slow operating speeds of the fatigue machine and the time-consuming aspects of the tests, extensive studies have not been made as yet on any other metals. One specimen each of rail steel as-rolled, rimmed structural steel, chromium-molybdenum alloy steel, ductile cast iron, and brass was tested at a stress somewhat higher than the yield strength of the individual materials. However, no failure had occurred in any of these materials after approximately 2,000,000 cycles of applied stress.

Supplementary Tests:

Several supplementary tests were run to determine the effect of stress concentrations on the strength of metals under repeated compression. A notched specimen of annealed rail steel, shown in Fig. 3(*c*), was tested at 75,000 psi. A small crack started in one of the notches after 1,000,000 cycles and, after 3,500,000 cycles, cracks were evident in both notches. However, neither of these cracks had spread appreciably after a total of 6,900,000 cycles. As shown in Table II no cracks developed in the unnotched specimens of this material, when it was subjected to a stress of 130,000 psi for 1,000,000 cycles.

Since the criterion for failure of the aluminum specimens was the first appearance of a crack, most tests of this metal were discontinued at this stage. Some tests were continued, however, to determine whether this crack would act as a stress raiser during subsequent cycles of stress. Aluminum specimen No. 7, in which a crack had formed after 6,443,000 applications of a stress of 52,000 psi, was tested for 3,313,000 additional cycles. The crack which had originally formed did not appear to propagate nor did new cracks develop.

Aluminum specimen No. 13 was tested under a maximum stress of 90,000 psi for 2,874,000 additional cycles of load after the initial crack had formed. In this case the crack propagated slowly around the entire circumference of the specimen and new cracks developed over most of the reduced section. The general direction of the cracks was horizontal, although the path was jagged and departed from the horizontal at various angles. During the latter stages of the test, pieces of the specimen were dislodged from regions adjacent to the crack. After 2,874,000 additional cycles of stress the specimen would no longer sustain the test load and therefore the test was discontinued. The specimen at this stage is shown in Fig. 10.

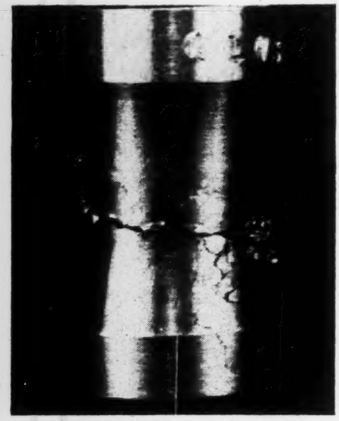

FIG. 10.—Fatigue Cracks in Aluminum Specimen No. 13, 2,874,000 Cycles After Appearance of First Crack.

SUMMARY

The work described herein is exploratory in nature and the only conclusion that can be drawn is that the endurance limit for all the materials tested appears to be considerably higher in axial compression than in axial tension or flexure. Moreover, for mild steel, the difference in strength between repeated compression and other types of repeated loads is greater than for the other metals. The low values of compressive endurance limit for steel reported by other investigators appear to have been the result of stress concentrations, residual stresses, or other phenomena disturbing the pattern of applied compressive stress.

Acknowledgment:

The work reported in this paper was part of the research program of the Civil Engineering Department of the University of Illinois and was sponsored by the Materials Branch of the Office of Naval Research under the general direction of N. M. Newmark, Research Professor of Structural Engineering.

Consultation and advice in developing the test program were obtained from H. F. Moore, Research Professor of Engineering Materials, Emeritus. Professor Moore offered many helpful suggestions throughout the work.

The tests were conducted by R. E. Elling and M. E. Hoffman, Research Assistants working under the direct supervision of W. H. Munse, Research Assistant Professor of Civil Engineering and R. J. Mosborg, Research Associate in Civil Engineering. The testing apparatus and the specimens were made in the shops of the Civil Engineering Department.

REFERENCES

(1) H. F. Moore, S. W. Lyon, and W. P. Inglis, "Tests of the Fatigue Strength of Cast Iron," *Bulletin No. 164*, Engineering Experiment Station, University of Illinois.

(2) T. Nishihara, and K. Kojima, "Diagram of Endurance Limit of Duralaumin for Repeated Tension and Compression," *Transactions*, Soc. Mechanical Engrs., Japan, Vol. 5, No. 20, August, 1939, pp. I-1 to I-2.

(3) T. Nishihara, and T. Sakurai, "Endurance Limit of Cast Iron for Repeated Tension and Compression," *Transactions*, Soc. Mechanical Engrs., Japan, Vol. 4, No. 15, May, 1938, pp. 105–110.

(4) T. Nishihara, and T. Sakurai, "Fatigue Strength of Steel for Repeated Tension and Compression," *Transactions*, Soc. Mechanical Engrs., Japan, Vol. 5, No. 18, February, 1939, pp. 25–29.

(5) Staff of the Battelle Memorial Institute. "Prevention of the Failure of Metals Under Repeated Stress," John Wiley and Sons, Inc., New York, N. Y., p. 135 (1941).

DISCUSSION

MR. R. E. PETERSON.[1]—The authors call attention to the fact that certain strength theories now being utilized for fatigue are not applicable for compressive conditions (left of AA, Fig. 11(a)).

Fatigue test data obtained in the region to the right of AA (Fig. 11(a)) seem to be in reasonable agreement with the shear-energy theory.[2, 3, 4, 5] In this area the mechanism of fatigue on a microscale is definitely due to shear. It would seem reasonable, however, that the crack propagation is influenced by normal stress. This is essentially the basis of the Mohr theory, where the envelope is a curve $\tau = f(\sigma)$. The Mohr theory, however, neglects the intermediate stress and consequently the diagram in Fig. 11 (light dashed lines) contains discontinuities. However, we can also postulate $\tau_0 = \phi(\sigma_0)$ where τ_0 and σ_0 are octahedral stresses (Fig. 11(b)).[6] If we assume τ_0 constant (dashed line, Fig. 11(b)) this corresponds to the shear-energy theory (dashed ellipse of Fig. 11(a)). If we assume $\tau_0 = \phi(\sigma_0)$ has the shape of the full line of Fig. 11(b) we obtain the full-lined figure of Fig. 11(a), which seems more realistic. This procedure, if it turns out to be sound, could be said to represent a modification of our present ideas, as contrasted to a radically new approach.

The use of shear theory raises an interesting question—if compression fatigue specimens are metallurgically polished and etched, are slip lines and micro cracks found under conditions comparable to those in tension fatigue specimens? There is good reason to expect slip lines in the compression specimens, and it is possible that these cyclically slipping surfaces might "abrade" into microcracks, which, however, might not spread into a gross crack. If so, would this condition be perfectly harmless or could this represent a hazard under shock conditions? In any event, it would be interesting to know if any observations of early slip or failure were made under high magnification.

MR. J. O. ALMEN.[7]—Messrs. Newmark, Mosborg, Munse, and Elling describe specimens that failed by fatigue from repeated compressive loads. I do not question the accuracy of their report insofar as it concerns the direction and magnitudes of the applied loads or the character of the observed failures. I do contend that they are wrong in intimating that their specimens failed from compressive stresses. They cannot express the load magnitudes in terms of pounds per square inch, and the measured load capacity applies only to the particular specimens used in the tests and not to the same metal in specimens of different form.

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[1] Manager, Mechanics Dept., Westinghouse Electric Corp., East Pittsburgh, Pa.

[2] H. Majors, B. D. Mills, and C. W. MacGregor, "Fatigue under Combines Pulsating Stresses," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 71, p. 269 (1942).

[3] R. E. Peterson, "Application of Stress Concentration Factors in Design," *Proceedings*, Soc. for Experimental Stress Analysis, Vol. 1, p. 118 (1943).

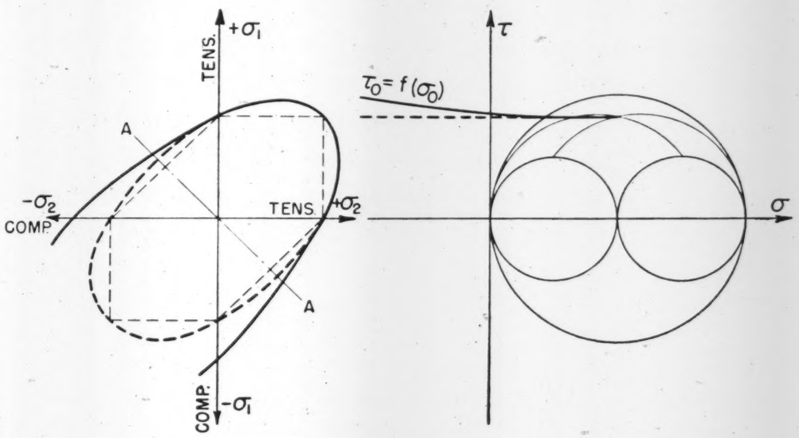
[4] J. Marin, "Interpretation of Experiments on Fatigue Strength of Metals Subjected to Combined Stresses," *Welding Journal*, Vol. 2, p. 245 (1942).

[5] C. R. Soderberg, "Handbook of Experimental Stress Analysis," p. 451, John Wiley and Sons Inc., New York, N. Y. (1950).

[6] A. Nadai, "Theory of Fracture and Flow in Solids," p. 94, 207, McGraw-Hill Book Co. Inc., New York, N. Y. (1950).

[7] Executive Consultant, Research Laboratory, Div., General Motors Corp., Detroit, Michigan.

The investigation failed to yield useful data because the tests and specimens were badly planned. Since only compressive loads were applied and since fatigue failures can develop only from tensile (macro) stresses, the report is incomplete until it shows first, how tensile stresses resulted from compressive loads and, second, the measured magnitudes of the tensile stresses causing failure. Inasmuch as these determinations were not and could not be made because of specimen design, the report as written is incomplete, inaccurate, and misleading.



(a) (b)

FIG. 11.—Conditions of Failure Under Bi-axial Stress.

Had the specimen design been such that residual stresses could have been measured and if such measurements had been made, it would have been found that residual tensile stresses normal to the plane of fracture developed in each specimen. Residual stresses of large magnitude will develop in initially stress-free specimens when subjected to repeated unidirectional loads because, under repeated loads, plastic yielding occurs at stresses far lower than the static yield stress of the material. Local plastic yielding develops residual stress of opposite sign to the stress causing plastic flow. Local plastic yielding under repeated compressive loads results in residual tensile stress. Because plastic yielding continues at a decreasing rate throughout the test, the magnitude of the resulting tensile stress also increases with load repetitions. It reaches its maximum magnitude when the applied load is at its minimum value.

An outer layer of a rod that is repeatedly loaded in compression will become residually stressed in tension because of greater plastic flow of the specimen surface. It is this residual tensile stress in the vulnerable surface metal that is responsible for fatigue failures in specimens that are nominally stressed only in compression. Since there are no external manifestations of such important changes in specimens undergoing tests except, perhaps, that the test machine must occasionally be adjusted to maintain the specified load, we are not aware of the presence of residual stresses nor can we know the direction of the stress causing failure unless adequate measurements are made.

The authors state that machine adjustments were found necessary to maintain the specified loads on their specimens but, even had the significance of these adjustments been recognized, the specimens used were not suitable for residual stress measurements. In the absence of data showing the approximate stress magnitude or even the stress sign, the only valid conclusion is that the procedure followed was incapable of providing the desired data.

MR. A. M. FREUDENTHAL.[8]—Mr. Almen has made an excellent point in distinguishing between the definition of the loading and the definition of the stress in fatigue tests.

We know very well that since no energy can be released by propagating a crack against a field of compressive stresses, a crack cannot propagate in such a stress field. Thus cracks that occur under compressive loading are probably cracks resulting from a previous deformation, which has set up a field of residual (tension or shear) stresses. Damage under compressive loading in fatigue is, therefore, probably not damage produced in loading but in unloading.

It would, therefore, be extremely important to define what we are talking about, if we are trying to discuss fatigue damage. We have been accustomed to talking about fatigue under tension, in which case the direction of the separation of particles on the submicroscopic scale coincides with the direction of the load. Under compression, the separation is opposite to the direction of the load which we apply, and therefore the conditions are somewhat unusual. We should, therefore, redefine our terms to represent the reality more correctly: fatigue due to loading in compression, for instance, is not identical with fatigue under compressive stress. And while the propagation of a fatigue crack under compressive stress may be impossible, propagation of such a crack under a "comprehensive load" can easily be visualized.

MR. S. A. GORDON.[9]—If the material presented was considered complete at this time, it would be indeed unfortunate, since it would only be of academic interest. A condition rarely occurs in airframe design where only an oscillating compression load exists. Consider a wing beam lower chord where the predominating loads are tension, but due to negative gusts and landing load, this lower chord also is subjected to cyclic compression loads, probably of lower magnitude.

Much more value could therefore be obtained from this study if the authors were to extend the investigation to determine what effect the accumulation of cyclic compressive stresses have on fatigue in tension.

MR. W. H. MUNSE (*author*).—I should like to point out that the work which has just been discussed is continuing.

The comments by Mr. Peterson proposing a modification in the shear-energy theory of failure to apply to these compressive fatigue tests is extremely interesting. However, it should be pointed out that the metallurgical studies which have been made to date indicate that the type of material and the eccentricity of loading are important factors in the development of axial compression fatigue cracks and must be taken into account in any theory of failure for this type of loading.

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[8] Professor of Civil Engineering, Columbia University, New York, N. Y.

[9] Technical Consultant, Battelle Memorial Institute, Columbus, Ohio.

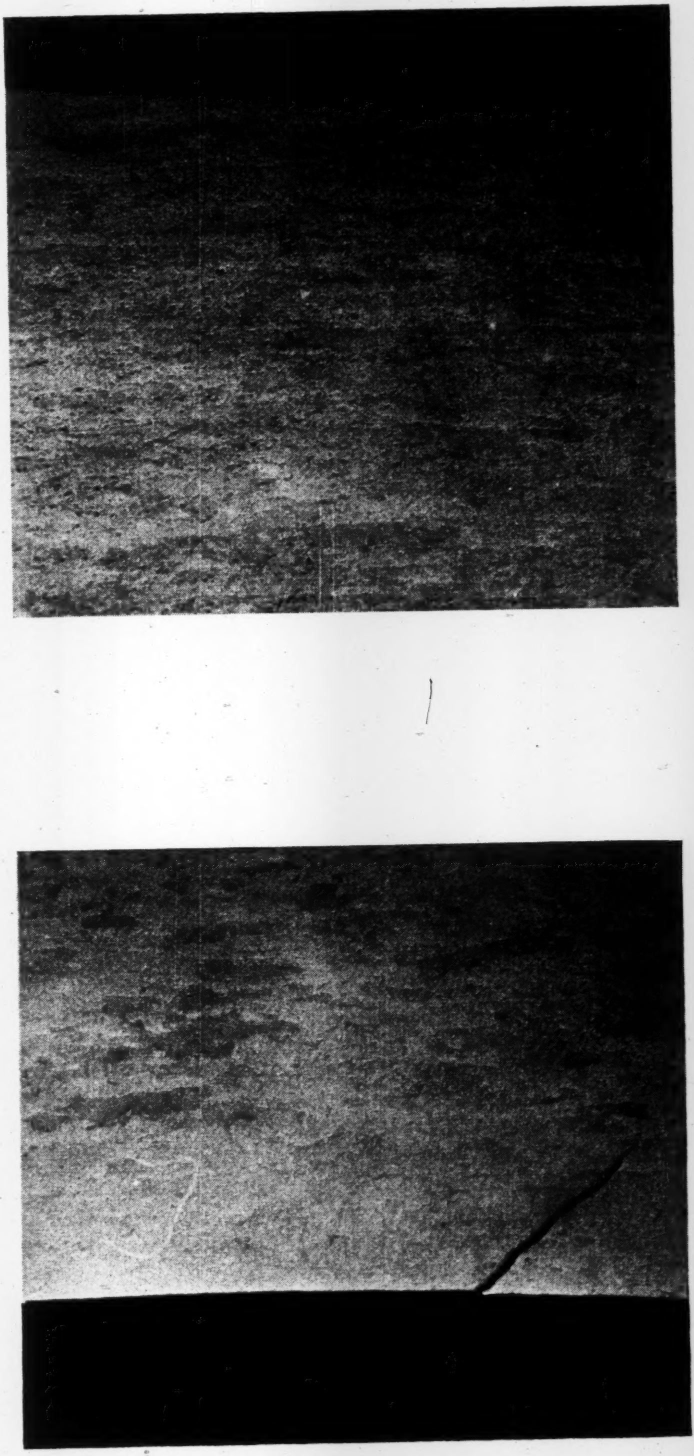

FIG. 12.—Cross-section of Aluminum Specimen No. 2 Showing Progression of Cracks from Opposite Surfaces, 35,000 Cycles after Appearance of First Crack (× 19).

Mr. Almen, in his discussion, questioned the stress distribution and the accuracy of the reported nominal stress in the axial compression specimens. We realize that the micro-stresses are not necessarily equal to the nominal stress, but we consider the nominal values of stress which we have reported to be the most reasonable values that can be reported. As stated in the paper, this investigation is primarily an exploratory study to provide basic data on compression fatigue and on the manner in which a compression fatigue failure might develop. It was not intended that the results of these tests be applied directly to other specimens and structures or parts of structures subjected to axial compression loadings.

Mr. Almen and Mr. Freudenthal state that tensile stresses are necessary to develop fatigue cracks. Sections of several of the cracked specimens, however, present a somewhat different picture. Figure 12, showing a section through an aluminum specimen, has very definite 45-deg cracks extending in from the surface of the specimen. Similar sections cut through steel specimens show horizontal breaks extending in from the surface of the specimen.

It might be pointed out, in answer to Mr. Almen's comments, that the strain measurements shown in Fig. 5 indicate that a slight tensile strain (macrostrain) did exist in the test specimens, but at the lower strain levels. This variation in strain across the specimen section, however, can be attributed to the slight eccentricity of loading indicated in Fig. 4. The strains measured at the three points on the surface of the specimen determine a strain plane which very closely checked the total load on the specimen, thereby indicating that the distribution of the strains across the section was linear. There may be residual stresses on the cross-section, but these are apparently different in steel and in aluminum, if they exist at all.

In closing I should like to reemphasize the fact that the tests reported in this paper are primarily exploratory. Nevertheless, because of the care taken in providing concentric loading to the specimens the tests have shown that some of the hypotheses previously offered in explanation of fatigue failures will not explain the results of repeated axial compression tests.

MR. N. M. NEWMARK (*author's closure by letter*).—I have read with great interest the comments by Messrs. Peterson, Almen, Freudenthal, and Gordon. In general all of the discussers are interested in the fundamental factors influencing failure. The purpose of our test program was to conduct fatigue tests under ranges of stress which had not hitherto been fully and carefully explored, so as to obtain a better basis for hypotheses as to the behavior of materials subjected to repeated loading.

The comments by Mr. Peterson are particularly pertinent. It is interesting to note that our results indicate a considerable influence of shear on failures even in axial-compression. The results of the tests of the aluminum alloy indicate a somewhat higher fatigue strength in repeated compression than in repeated tension, but there is indicated also, in Fig. 12, a fairly definite shear crack, or at least a crack at a 45-deg slope from the direction of stress, in a cross-section of the aluminum alloy specimen. In steel this same type of influence was not observed. In general, the cracks that were obtained in steel were perpendicular to the direction of loading, and the fatigue strengths or endurance limits, if we can believe that we have actually obtained these, are very much greater than the corresponding values for tests in repeated tension.

As a matter of fact, the more carefully we are able to align our specimens, the higher is the stress to which we can subject the specimen, for several millions of cycles of loading, without causing any crack.

It is likely that as soon as cracks of any material size appear in the specimen, the distribution of stress over the specimen no longer can remain approximately uniform, and consequently it is possible for the crack to spread in about the same way that a crack is initiated and propagates in a notched specimen subjected to axial-compression. However, the spread of a crack once initiated is not rapid in a compression field. One must be fairly careful in drawing conclusions about the propagation of cracks under conditions of nominally uniform stress distribution since such a distribution must change with the configuration of the specimen.

Mr. Almen is severely critical of the paper. However, it appears to us that his criticism is equally applicable to all fatigue test data whether it is in tension, bending, or compression. It is the usual practice in referring to fatigue test results to report the nominal stress. This is certainly done in axial-tension tests and it is commonly done in tests in flexure. It is certainly wrong to consider that the nominal stress has any significance in specimens that are notched or that have severe stress concentrations. However, there is possibly a greater significance of the nominal stress in a specimen which has a reasonably uniform distribution of stress over its entire area. We are certainly aware of the fact that the stress distribution from point to point or crystal to crystal even in a so-called uniform stress field, may show variations because of differences in microstructure and in local irregularities in the material. However, it can be contended that these differences and microstresses are of no greater significance in axial-compression than they are in other types of stress distribution.

The authors object to Mr. Almen's statement, that "it would have been found that residual tensile stresses normal to the plane of fracture developed in each specimen." This is an unwarranted assumption. It requires that in aluminum the residual tensile stresses develop at an angle of 45 deg with the direction in which they develop in steel. We are continuing our work and expect to have some data to report on this point later. We prefer to wait until the test data are available before making any further comment.

One cannot avoid the conclusion from these test results that in so-called 'uniform" stress fields, the fatigue strength under repeated compressive loads is considerably greater than under repeated tensile loads for some materials and not so much greater for others. However, there appears to be some definite influence of the shear stress on the formation of cracks even in repeated compression for some of the materials.

Previously reported results of tests in compression were even more "incomplete, inaccurate, and misleading" than those given in our paper. There have been several series of tests which have been reported in the literature. Some of these gave indications that the strength in repeated compression was of about the same order of magnitude as that in repeated tension, apparently because the stress distribution in the specimens reflected the influence of non-uniform distributions of stress. In our own tests, where some bending was observed in the specimens, we generally obtained cracks at a much lower number of cycles than was the case where more nearly uniform distributions were achieved. As a result of improvements in our apparatus, we now have increased the accuracy of alignment and have obtained corresponding increases in fatigue strength under compressive loading for the steel specimens.

We hope to have soon some information to indicate whether Mr. Almen's statement is true that an outer layer of a rod which is repeatedly loaded in compression will become residually stressed in tension. This does not appear entirely reasonable. At any rate an investigation can be made of this point by boring out the interior of a specimen which has been previously loaded repeatedly in compression and we are making plans to do this. It is more likely that local variations in the properties of the material can cause localized tensile stresses. However, it is not necessary that fatigue cracks are caused only by tensile stresses. This, it appears to us, is only an assumption regarding the nature of fatigue failure.

The same general comments apply to Mr. Freudenthal's discussion. A crack cannot propagate in a field in which uniform compressive stresses exist in all directions. However, there seems to be no reason why a crack cannot propagate in a field in which shearing stresses are present, and such a field is obtained when one has uniaxial-compression. In any event, if our tests have the effect of causing a redefinition of the terms used in discussing fatigue damage, our program has served a most useful purpose.

Our program reported certain test data. The interpretation of these data was not a part of the paper which we presented. The facts presented cannot be denied. What is required is a better theory or hypothesis as to the nature of fatigue damage, which is capable of explaining these results. Incidentally, it was not the intention of the authors to indicate that fatigue failures were due to local compressive stresses. The authors were not naive enough to believe that the compressive stresses over the cross-section of the specimen were entirely uniform. Any non-uniformity might have produced local residual tensile stresses.

Mr. Gordon raises an important practical point. Our future work will be devoted in part toward answering questions similar to those which he has raised, namely, the effect of ranges of stress from compression to tension with as nearly a uniform distribution of stress over the cross-section as it is possible to achieve.

PREDICTION OF RELAXATION OF METALS FROM CREEP DATA*

By Irving Roberts[1]

Synopsis

In recent years, evidence has been accumulating that relaxation rates of metals can be predicted from tension creep data by means of the strain-hardening assumption. This has been accompanied by considerable controversy, and the present paper presents a theoretical discussion which should help to clarify some of the confusion which has existed in this field. Following this, a study of experimental data is given, showing that the strain-hardening assumption yields accurate results not only for copper, but also for carbon steel and for the alloy S-816.

While an adequate fundamental theory of creep has not yet been developed, there must exist, for any given metal at a fixed temperature, a law which expresses the creep rate, $d\epsilon_p/dt$, as a function of one or more of the variables, stress, σ, plastic strain, ϵ_p, and time, t. Both creep measurements and relaxation measurements give us only the results of integration of such a differential law. In the case of creep data, where stress is held constant, the integration has the limitation of constant σ, while for relaxation data, where the total strain is held constant, the integration has the more involved restriction that

$$\epsilon_p + \frac{\sigma}{E} = \epsilon^* = \text{constant} \quad \ldots\ldots (1)$$

For the equations in this paper the following nomenclature is used:

C, K = constants,
E = modulus of elasticity, psi,
L = length of bar, in.,
S = $K\,(e^{\sigma/s} - 1)$,
m, n, p, q, s = constants,
t = time, hr,
ϵ^* = initial elastic strain, dimensionless,
ϵ_p = plastic strain, dimensionless,
σ = stress, psi, and
σ_0 = initial stress, psi.

In spite of these limitations, such data may be used to determine empirically the nature of the differential creep law. Constant-stress creep data lend themselves most readily to such analysis, while relaxation experiments may be used as a check on the accuracy of the differential law so obtained.

As an example for purposes of this discussion, we will assume a family of creep curves, each of which is linear on log-log paper, so that each curve may be expressed by the equation

$$\epsilon_p = Ct^m \quad \ldots\ldots\ldots\ldots (2)$$

in which the constant, m, is positive and less than one. Furthermore, it will be assumed that these curves are related to each other by the function of stress,

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($e^{\sigma/s} - 1$), so that the entire family is given by the equation

$$\epsilon_p = K(e^{\sigma/s} - 1)t^m \quad \ldots\ldots\ldots (3)$$

This is the relation first proposed by Soderberg (1)[2] and found by Popov (2) to give a fair representation of the creep data of Davis (3) on copper at 165 C. Many other empirical creep relations have been proposed, and undoubtedly an infinite variety of them is possible.

There are pitfalls in the use of Eq 3, or any other constant-stress creep relation, which must be avoided. The equation says that there exists a single value of ϵ_p for each value of σ and each value of t. In actual fact, ϵ_p is not a single-valued function of σ and t. By changing the stress history of the sample during the creep process, many values of ϵ_p can be made to occur at the same values of σ and t. A simple example of this is shown in Fig. 1, in which changes in stress have been made in the upper and lower creep curves. The three curves give three different values of ϵ_p at identical values of σ and t, namely, σ_2 and t_1. However, the substitution of the values σ_2 and t_1 into Eq 3 can give only one value of ϵ_p.

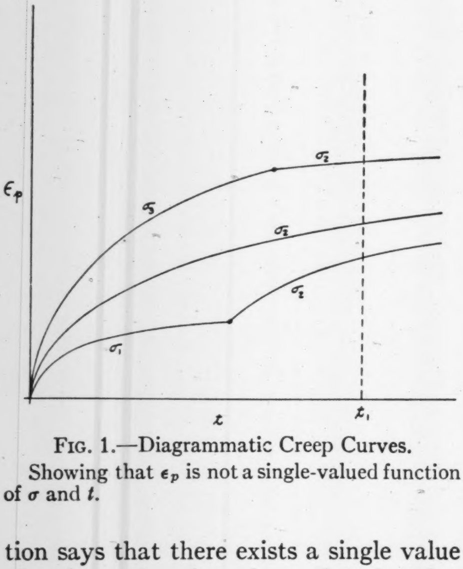

FIG. 1.—Diagrammatic Creep Curves.

Showing that ϵ_p is not a single-valued function of σ and t.

Thus, Eq 3 is incorrect for any creep curve except that determined at constant stress and starting at zero time and zero ϵ_p. The equation cannot predict the effect of changes in σ at values of t greater than zero. However, the equation does possess all the requirements necessary for it to be the result of integration at constant σ of an unknown differential creep law between the time limits 0 and t, and between the strain limits 0 and ϵ_p.

To obtain the differential creep law, we carry out the reverse process, that is, we differentiate Eq 3 at constant σ. However, this yields an infinite number of solutions. For example, direct differentiation of Eq 3 gives

$$\frac{d\epsilon_p}{dt} = mK(e^{\sigma/s} - 1)t^{m-1} \quad \ldots\ldots (4)$$

If we take the mth root of both sides of Eq 3 and then differentiate, we obtain

$$\frac{d\epsilon_p}{dt} = \frac{m[K(e^{\sigma/s} - 1)]^{1/m}}{\epsilon_p^{1/m-1}} \quad \ldots\ldots (5)$$

Or, if we square both sides of Eq 3 and differentiate, we obtain

$$\frac{d\epsilon_p}{dt} = \frac{m[K(e^{\sigma/s} - 1)]^2 t^{2m-1}}{\epsilon_p} \quad \ldots\ldots (6)$$

One might argue that Eqs 4, 5, and 6 are really identical, since the substitution for ϵ_p or t from Eq 3 into the right-hand sides of Eqs 4, 5, and 6 will yield identical results. While one might be accustomed to making such a substitution in other problems, it is not allowable in this case because of the defective nature of Eq 3. Equation 3 is an incorrect relationship in general, and only becomes correct when we make the limitation of constant σ and start each creep curve at the origin. The substitution would be allowable if we had written the partial derivative $(\partial\epsilon_p/\partial t)_\sigma$ instead of $d\epsilon_p/dt$, and if we would always perform the integration of these differential equations between the limits 0 and t and between 0 and ϵ_p. These limitations, would, of course, make the result trivial, since we are interested in setting up an equation such as Eqs 4, 5, or 6 as a general law of creep which can be integrated between any limits and in which σ is an independent variable.

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[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 825

That these equations are different in general can be seen, for example, from the fact that their integration at constant σ between the limits t_1 and t_2 and the limits ϵ_{p_1} and ϵ_{p_2} yields different results. Performing this integration of Eq 4 gives

$$\epsilon_{p_2} - \epsilon_{p_1} = K(e^{\sigma/s} - 1)(t_2^m - t_1^m) \quad \text{..(7)}$$

while Eq 5 gives

$$\epsilon_{p_2}^{1/m} - \epsilon_{p_1}^{1/m} = [K(e^{\sigma/s} - 1)]^{1/m}(t_2 - t_1) \quad \text{..(8)}$$

and Eq 6 gives

$$\epsilon_{p_2}^2 - \epsilon_{p_1}^2 = [K(e^{\sigma/s} - 1)]^2(t_2^{2m} - t_1^{2m}) \quad \text{..(9)}$$

Equations 7, 8, and 9 are not identical. Likewise, combination of Eqs 4, 5, and 6 with the condition of relaxation, Eq 1, followed by integration to obtain σ as a function of time, yields entirely different results. Thus, Eqs 4, 5, and 6 are different, but all three equations, and many others, have the property of integrating at constant σ, between the limits 0 and t and the limits 0 and ϵ_p, to give Eq 3.

The problem is now to choose the correct one of this infinite number of possible differential equations. One possible choice may be made by the assumption of "time-hardening," that is, that the creep rate is a function of the variables stress and time only. This limits the selection to Eq 4, since it is apparently true that this is the only equation in which σ and t are the only independent variables and which will integrate to give Eq 3. In like manner, we may make the assumption of "strain-hardening," that is, that the creep rate is a function of the variables stress and plastic strain only, and this limits the choice to Eq 5. Finally, we may assume that the creep rate is a function of all three variables, stress, plastic strain, and time, and, in this case, there are an infinite number of possible differential equations.

Soderberg (1) obtained a differential equation by differentiating Eq 3, not at constant σ, but with σ variable. While not expressed in the same form in Soderberg's paper, the result of the differentiation is

$$d\epsilon_p = K\left[t^m \frac{e^{\sigma/s}}{s}\, d\sigma + (e^{\sigma/s} - 1)mt^{m-1}dt\right] \quad \text{..(10)}$$

This equation cannot be correct because the differentiation implies that Eq 3 is correct when σ is allowed to vary, and, as has been shown above, this is not the case. Stating this in another way, the integration of Eq 10 between the limits 0 and t and the limits 0 and ϵ_p will give Eq 3, not only with constant σ, but also with variable σ. The writer has previously shown (4) that the use of Eq 10 with Eq 1 to solve the relaxation problem yields a result which is identical with that obtained by simple algebraic solution of Eqs 3 and 1 simultaneously. This again demonstrates that Eq 3 has been implicitly assumed to be correct with σ continuously variable.

A similar argument to show the faulty nature of Soderberg's method was given by Davenport (5) in his discussion of Soderberg's paper. This point is being brought up again here only because Popov (2) has apparently found good agreement in using Soderberg's method to predict the relaxation of copper. As will be seen in the body of this paper, the conclusion that Soderberg's method gives good agreement is in error.

COPPER

Alternate Load Experiment:

An experiment by Phillips and Smith (6), later confirmed by Davenport (7), provides one of the earliest pieces of evidence in favor of the strain-hardening assumption. In this experiment, tests were made on copper with alternate loads of 10 and 70 per cent of the tensile strength for equal periods of time, and it was found that the total plastic strain was independent of the order of applying the loads. A diagrammatic representation of this experiment for two equal periods is shown in Fig. 2. In the upper curve, a sample is held at stress σ_2 for a period t_1 during which the plastic strain increases from 0 to ϵ_{p_2}. The stress is then reduced to σ_1 for an additional period t_1, and the plastic strain increases from ϵ_{p_2} to ϵ_{p_3}. In the lower curve, a sample is held at the lower stress σ_1 for the period t_1, and the plastic strain increases from 0 to ϵ_{p_1}. Then the stress is increased to σ_2 and held for an additional period t_1, and, according to the stated result, the final strain ϵ_{p_3} is identical with that obtained with the sample giving the upper curve.

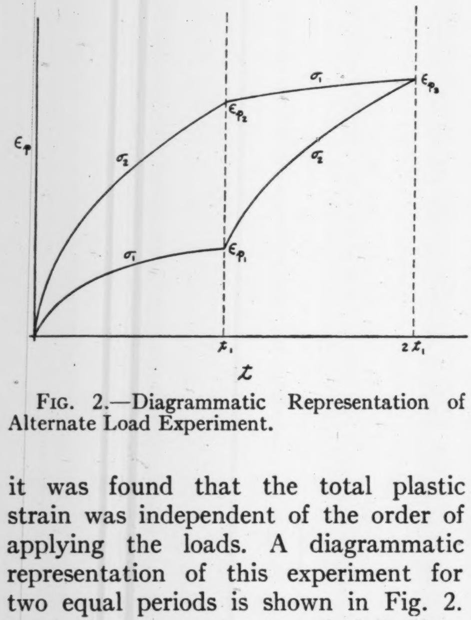

FIG. 2.—Diagrammatic Representation of Alternate Load Experiment.

Davenport (7) used this experiment to disprove time-hardening, but the argument can be carried further; we can show that this result is in accord with strain-hardening. To show this, let us assume that the creep data for the samples of copper used in the above experiments can be represented by Eq 3. A similar argument will apply for other empirical creep equations, and Eq 3 is used here only as a convenient example. The differential equation for the strain-hardening assumption is therefore Eq 5, and the result of integration at constant σ between general limits is Eq 8. Letting the expressions $K(e^{\sigma_1/s} - 1)$ and $K(e^{\sigma_2/s} - 1)$ be denoted by S_1 and S_2 for convenience, we may write for the upper curve of Fig. 2,

$$\epsilon_{p_2}^{1/m} = S_2^{1/m} t_1 \qquad \text{(11)}$$

$$\epsilon_{p_3}^{1/m} - \epsilon_{p_2}^{1/m} = S_1^{1/m}(2t_1 - t_1) \qquad \text{(12)}$$

Adding these two equations,

$$\epsilon_{p_3}^{1/m} = (S_1^{1/m} + S_2^{1/m})t_1 \qquad \text{(13)}$$

For the lower curve of Fig. 2, we have

$$\epsilon_{p_1}^{1/m} = S_1^{1/m} t_1 \qquad \text{(14)}$$

$$\epsilon_{p_3}^{1/m} - \epsilon_{p_1}^{1/m} = S_2^{1/m}(2t_1 - t_1) \qquad \text{(15)}$$

Adding Eq 14 and 15,

$$\epsilon_{p_3}^{1/m} = (S_1^{1/m} + S_2^{1/m})t_1 \qquad \text{(16)}$$

The identity of Eqs 13 and 16 shows that the strain-hardening assumption predicts identical values of ϵ_{p_3} by both paths.

To test the time-hardening assumption, we make use of the integrated form, Eq 7. For the upper curve of Fig. 2, we write

$$\epsilon_{p_2} = S_2 t_1^m \qquad \text{(17)}$$

$$\epsilon_{p_3} - \epsilon_{p_2} = S_1[(2t_1)^m - t_1^m] \qquad \text{(18)}$$

Adding, we obtain

$$\epsilon_{p3} = [S_1(2^m - 1) + S_2]t_1^m \;\ldots\ldots (19)$$

For the lower curve of Fig. 2, we have

$$\epsilon_{p1} = S_1 t_1^m \;\ldots\ldots\ldots\ldots (20)$$

$$\epsilon_{p3} - \epsilon_{p1} = S_2[(2t_1)^m - t_1^m] \;\ldots\ldots (21)$$

and adding, we obtain

$$\epsilon_{p3} = [S_1 + S_2(2^m - 1)]t_1^m \;\ldots\ldots (22)$$

Equations 19 and 22 are not identical. By similar reasoning, it can be shown that integrated forms of the type of Eq 9, which result from the many differential equations which express $d\epsilon_p/dt$ as a function of all three variables σ, ϵ_p and t, do not give the observed result. It can also be shown that the strain-hardening assumption gives the correct result for more than two equal subdivisions of time, and that the final value of ϵ_p is independent of the number of subdivisions (that is, independent of the number of times the load is alternated), so long as the total time for each load is the same.

Relaxation at Room Temperature:

Davis (3) has reported a series of creep and relaxation tests on commercial copper at room temperature. The creep data gave straight lines on log-log paper for tests lasting about a year, and the individual creep curves could be related to each other by a simple exponential stress function. A differential equation for strain-hardening was written, of the form

$$\frac{d\epsilon_p}{dt} = K\frac{\sigma^q}{\epsilon_p^p} \;\ldots\ldots\ldots\ldots (23)$$

corresponding to the experimental creep equation

$$\epsilon_p = K_1 \sigma^{q/(p+1)} t^{1/(p+1)} \;\ldots\ldots (24)$$

where p and q were found to be approximately 5 and 7, respectively.

Combining Eq 23 with Eq 1, and with

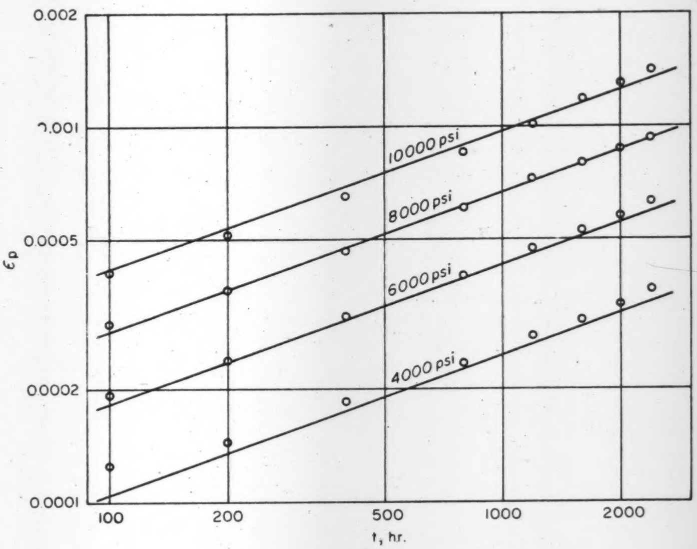

FIG. 3.—Creep of Copper at 165 C.

Data points from Davis (3). Curves calculated from the equation of Popov (2)

$$\epsilon_p = 26.8 \times 10^{-6} \left(e^{\sigma/7500} - 1\right) t^{0.372}.$$

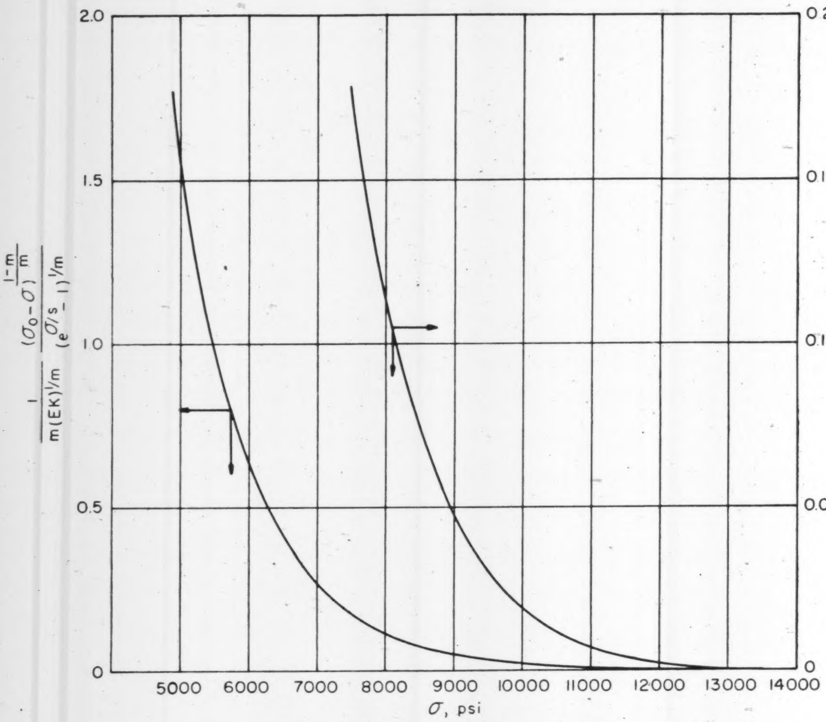

FIG. 4.—Relaxation of Copper at 165 C.

Graphical integration of Eq 29 for σ_0=13,500 psi.

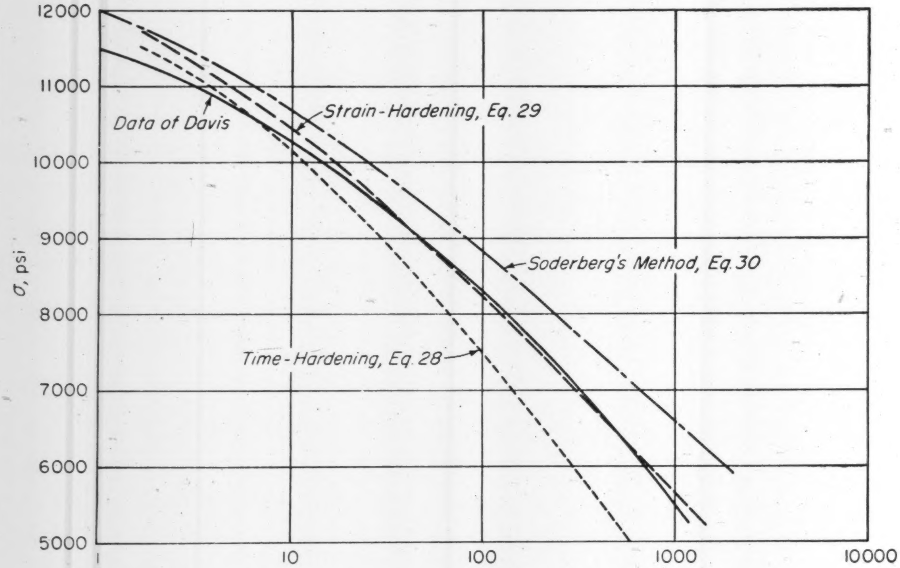

FIG. 5.—Relaxation of Copper at 165 C. σ_0 = 13,500 psi.

Solid curve gives test data of Davis (3).

the differentiated form of Eq 1, namely,

$$\frac{d\epsilon_p}{dt} = -\frac{1}{E}\frac{d\sigma}{dt} \ldots\ldots\ldots\ldots (25)$$

Davis obtained the following form of equation for relaxation

$$t = -C\int_{\sigma_0}^{\sigma} \frac{(\sigma_0 - \sigma)^p}{\sigma^q}\, d\sigma \ldots\ldots (26)$$

in which σ_0 is the initial stress in the relaxation experiment. Since p and q are integers, Eq 26 was integrated directly. The integrated result of Eq 26 showed excellent agreement with the relaxation data over a range of stress from about 4000 to 22,000 psi and over a range of time from about 0.1 to 1000 hr.

In spite of the excellent agreement, Davis objected to this theory, that is, Eq 23, on the basis that it predicts an infinite strain rate at the beginning of a creep test. It is difficult to see any fundamental reason why this should not be the case, and, in any event, the infinite strain rate only occurs for an infinitesimal period of time.

Relaxation at 165 C.:

Davis did not apply the above theory, which worked so well at room temperature, to his higher temperature data "where a minimum creep rate is observed." Presumably, this means that the creep curves approached straight lines on ordinary coordinate paper and, therefore, were curved on log-log paper. However, Popov (2) fitted an empirical equation to Davis' data of the form of Eq 3 and simply neglected the curvature. A plot of the creep data read from Davis' curves and of the straight lines predicted from Popov's equation is given in Fig. 3. Apparently, the deviation of the calculated from the observed curves is not important, since Popov was able to use this equation to solve the relaxation problem with good accuracy.

In testing the time-hardening assumption, Popov used a step-by-step numerical procedure which is, in effect, equivalent to the solution of Eq 4 on the basis of finite rather than infinitesimal intervals. The labor and possible inaccuracy of this method are unnecessary, since Eq 4 can be solved analytically for the conditions of relaxation. Combining Eq 4 with Eq 25, we obtain the equation

$$\int_{\sigma_0}^{\sigma} \frac{d\sigma}{e^{\sigma/s} - 1} = -mEK\int_0^t t^{m-1}\, dt \ldots (27)$$

which upon integration gives

$$t = \left[\frac{s}{EK}\ln\frac{1 - 1/e^{\sigma_0/s}}{1 - 1/e^{\sigma/s}}\right]^{1/m} \ldots\ldots (28)$$

Substituting the values of the constants

$s = 7500$
$E = 14.1 \times 10^6$
$K = 26.8 \times 10^{-6}$
$m = 0.372$

and solving for the case of $\sigma_0 = 13{,}500$ psi gives the lower curve of Fig. 5.

In testing the strain-hardening assumption, Popov again used a step-by-step numerical method and also the method of Davenport (7) which involves the reading of slopes from the creep curves. Here again, an analytical solution is possible. Combining Eq 5 with Eqs 1 and 25, and remembering that $\epsilon^* = \sigma_0/E$, we obtain

$$t = -\int_{\sigma_0}^{\sigma} \frac{1}{m(EK)^{1/m}} \frac{(\sigma_0 - \sigma)^{1-m/m}}{(e^{\sigma/s} - 1)^{1/m}}\, d\sigma \ldots (29)$$

Unfortunately, we have been unable to integrate this equation in terms of simple functions and have resorted to graphical integration. Figure 4 gives a plot of the function within the integral sign against σ for the case of $\sigma_0 = 13{,}500$ psi. The area under this curve gives the time directly. A portion of the curve is also plotted on a 1:10 ordinate scale in order to increase the accuracy of area determination in the higher stress region. The results of this graphical integration are plotted in Fig. 5.

In testing the validity of Soderberg's method, Popov used the stepwise numerical solution proposed by Soderberg. This is no longer necessary since it has been shown (4) that the identical result is obtained by simultaneous solution of Eqs 1 and 3, giving

$$t = \left[\frac{\sigma_0 - \sigma}{KE(e^{\sigma/s} - 1)} \right]^{1/m} \quad \ldots\ldots (30)$$

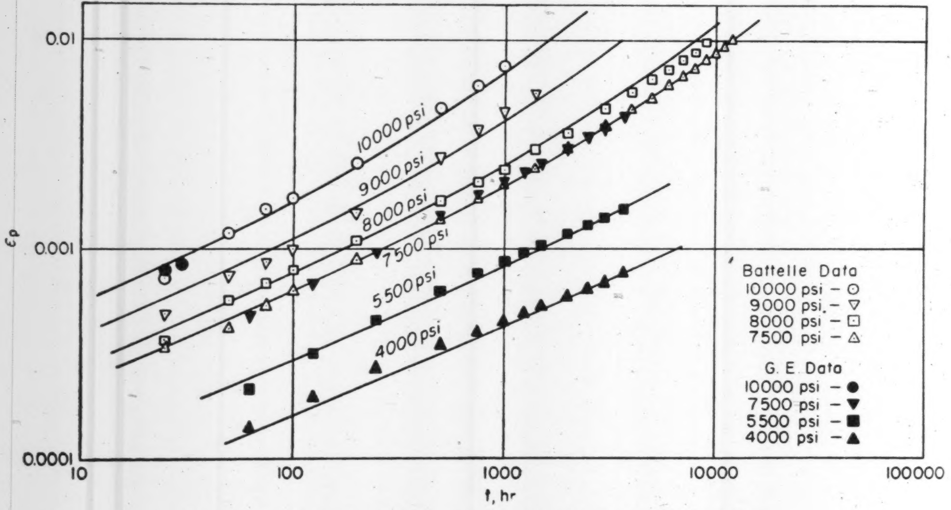

FIG. 6.—Creep of 0.35 per cent Carbon Steel K-20 at 850 F.
Curves calculated from Eq 31.

Substituting for the constants their values given above, and solving at $\sigma_0 = 13{,}500$ psi, gives the upper curve of Fig. 5.

In accord with Popov's findings, Fig. 5 shows that the strain-hardening calculation gives good agreement with the test data and that the time-hardening calculation predicts a curve which falls below the data. In the case of Soderberg's method, however, the deviation from the test data is almost as great as for time-hardening, whereas Popov finds Soderberg's method to give substantial agreement. Examination of Fig. 4 of Popov's paper reveals that the calculated Soderberg curve lies 200 to 350 psi below the curve given here. It is possible that this error was caused by the use of the stepwise numerical method. In addition, the test data of Davis appear to have been plotted incorrectly in Fig. 4 of Popov's paper, the curve being about 350 psi too high at 1000 hr, although the data seem to have been plotted accurately in Fig. 5 of Popov's paper. These two errors, both in the direction of reducing the difference between the test curve and the Soderberg curve, appear to account for the discrepancy. It may be noted here that for the other examples given in this paper, Soderberg's method shows similar disagreement with the data.

CARBON STEEL

During the years 1936 through 1948, under the auspices of the ASTM-ASME Joint Committee on the Effect of Temperature on the Properties of

Metals, there appeared a large number of creep and relaxation measurements at 850 F on a 0.35 per cent carbon steel, designated as K-20. These data afford an unusual opportunity for comparison of creep and relaxation phenomena, since the creep data cover a wide range, and since a variety of relaxation experiments was reported.

Creep Data:

The bulk of the creep data was obtained at Battelle Memorial Inst. and at the General Electric Co. The Battelle data were reported by Cross and co-workers (8, 9), and the General Electric data were reported by Robinson (10). These results have also been summarized in the compilation of creep data published by the Joint Committee in 1938 (11). Figure 6 gives a log-log plot of the data. In most cases, the points were taken directly from the data tables, but, in order to improve the accuracy of the curves, additional points were read from the published graphs wherever this was found desirable. In the references, the results were given as total strain, and the measured initial elastic strain was not always in accord with the measured value of the modulus, 20×10^6 (12). For such results, it was assumed that only the initial strain measurement was in error, and the plastic strains were obtained by deducting the calculated elastic strain from the total strain figures. This assumption had the effect of making the 7500 psi data of both laboratories coincide remarkably well, as may be seen in Fig. 6.

In fitting an empirical equation to the data of Fig. 6, it was felt that the greatest weight should be placed on the 7500-psi curve, since this covers the largest range and appears to be the best known. Furthermore, the equation could not be of the simple logarithmic

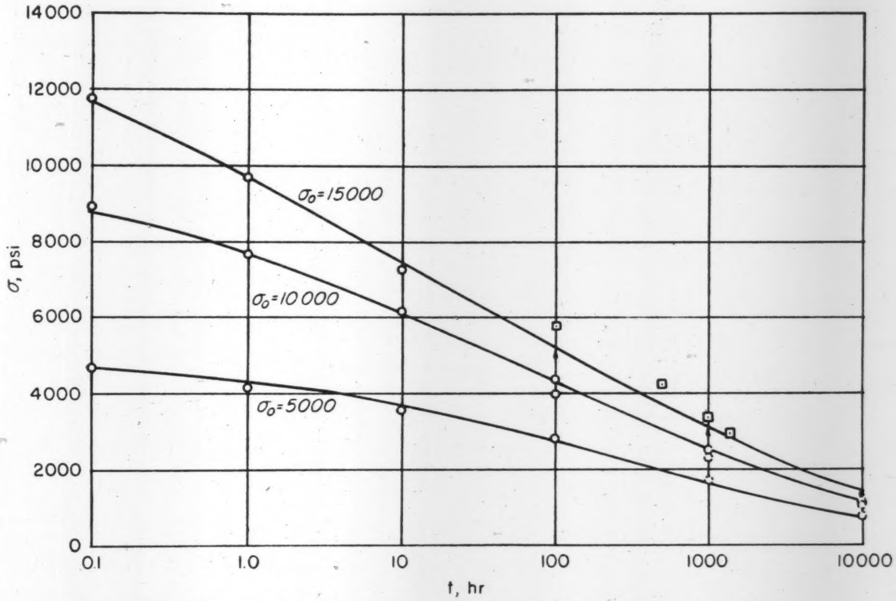

FIG. 7.—Relaxation of 0.35 per cent Carbon Steel K-20 at 850 F.

Circled data points from Boyd (13). Squares are data of U. S. Naval Engineering Experiment Station for σ_0= 15,000 psi. (14). Curves calculated from Eq 33.

type, since the creep data show a definite curvature, particularly at the higher stresses. Finally, in setting up a more involved equation, it was felt that it should be explicit in t rather than in ϵ_p, since this results in simpler algebraic operations in derivation of the differential equation for strain-hardening.

The equation finally developed is

$$t = \left[\frac{\epsilon_p}{K(e^{\sigma/s} - 1)}\right]^n \frac{1}{(1 + q\epsilon_p)^m} \quad \text{..(31)}$$

in which the values of the constants are

$K = 1.176 \times 10^{-5}$
$s = 3500$
$q = 489$
$n = 2.55$
$m = 1.5$

It will be noted that if the constant q were equal to zero, Eq 31 would reduce to Eq 3. Therefore, the term $(1 + q\,\epsilon_p)^m$ may be considered as a correction term to introduce the required curvature on log-log paper. The solid curves in Fig. 6 were calculated from Eq 31.

Relaxation Data:

Boyd (13) has reported the results of a series of tests in which the total strain of the sample was automatically held constant, thus duplicating accurately the condition of Eq 1. The data were presented by Boyd in the form of a cross-plot giving the residual stress at various times as a function of initial stress. Actual data points were given over the range of time from 0.1 to 100 hr, but extrapolated values were also given at 1000 and at 10,000 hr. It is presumed that the extrapolation was done by the empirical method of Robinson (12). The data for initial stresses of 5000, 10,000, and 15,000 psi are plotted in Fig. 7. Also shown in Fig. 7 are data for $\sigma_0 = 15{,}000$ psi obtained at the U. S. Naval Engineering Experiment Station and reported in Table IX of the 1948 report of the Joint Committee (14).

To predict these data by the strain-hardening assumption, we differentiate Eq 31 to give the following as the differential law of creep:

$$\frac{d\epsilon_p}{dt} = \frac{[K(e^{\sigma/s} - 1)]^n}{\dfrac{n\epsilon_p^{n-1}}{(1 + q\epsilon_p)^m} - \dfrac{mq\epsilon_p^n}{(1 + q\epsilon_p)^{m+1}}} \quad \text{..(32)}$$

Combining with Eqs 1 and 25, we obtain

$$t = -\int_{\sigma_0}^{\sigma} \frac{1}{E\left[1 + q\left(\dfrac{\sigma_0 - \sigma}{E}\right)\right]^m} \cdot \left[\frac{\left(\dfrac{\sigma_0 - \sigma}{E}\right)}{K(e^{\sigma/s} - 1)}\right]^n \cdot \left[\frac{n}{\left(\dfrac{\sigma_0 - \sigma}{E}\right)} - \frac{mq}{1 + q\left(\dfrac{\sigma_0 - \sigma}{E}\right)}\right] d\sigma \quad \text{..(33)}$$

This equation was integrated graphically, in a manner similar to the integration of Eq 29, and the results are the solid curves of Fig. 7. Over the range of the data, the agreement is excellent. It is also of interest to note that the extrapolated 1000 and 10,000-hr points show that the empirical method of extrapolation predicts the same general shape of relaxation curve as is given by Eq 33.

Robinson's Step-Down Test:

The Robinson step-down test on K-20 carbon steel was a simulated relaxation test (10, 11) in which a sample was held at an initial stress of 10,000 psi until a total strain of 0.0015 was reached, after which the stress was reduced in steps to maintain the total strain approximately constant. To predict the results of each constant stress period in such an experiment, we integrate Eq 32 at constant σ between the limits t_1 and t_2 and the limits ϵ_{p_1} and ϵ_{p_2}. The result of this integration is

$$\frac{\epsilon_{p_2}^n}{(1 + q\epsilon_{p_2})^m} - \frac{\epsilon_{p_1}^n}{(1 + q\epsilon_{p_1})^m} = [K(e^{\sigma/s} - 1)]^n(t_2 - t_1) \quad \text{....(34)}$$

In Table I, the first four columns give, for each of the constant stress periods, the stress, the time interval, the initial plastic strain, and the change in plastic strain observed during the period. The fifth column gives the change in plastic strain ($\epsilon_{p_2} - \epsilon_{p_1}$) calculated from Eq 34 using the observed value of ϵ_{p_1} for each step. The last column shows the difference between the observed and the calculated increments of plastic strain. The agreement is considered satisfactory in view of the probable error in the measurements.

TABLE I.—STEP-DOWN TEST ON K-20 CARBON STEEL AT 850 F.

σ, psi	$t_2 - t_1$, hr	ϵ_{p1} (observed)	$\Delta\epsilon_p$ (observed)	$\Delta\epsilon_p$ (calculated)	Deviation, $\Delta\epsilon_p$
10 000.......	30	0.00000	0.00085	0.00091	+0.00006
7 500.......	88	0.00085	0.00019	0.00017	−0.00002
5 500.......	172	0.00104	0.00010	0.00009	−0.00001
4 000.......	935	0.00114	0.00011	0.00006	−0.00005
3 000.......	2475	0.00125	0.000075	0.00005	−0.000025

The Crane "b = 5" Test:

In this experiment, also reported by Robinson (**12**), the length of the test bar was held constant, but only one-fifth of its length was heated to 850 F. Thus, in such a relaxation experiment, after a given time, the amount of plastic strain is approximately five times the decrease in elastic strain. This relationship is only approximate because the elastic modulus of the unheated and heated lengths are different. If the heated length is denoted by L, and if E_1 and E_2 are the values of the modulus at 850 F. and at room temperature, respectively, we may write

$$L\left(1 + \epsilon_p + \frac{\sigma}{E_1}\right) + 4L\left(1 + \frac{\sigma}{E_2}\right) = \text{constant} = L\left(1 + \frac{\sigma_0}{E_1}\right) + 4L\left(1 + \frac{\sigma_0}{E_2}\right) \quad \text{....(35)}$$

where σ_0 is the initial stress. Rearranging,

$$\epsilon_p = (\sigma_0 - \sigma)\left(\frac{1}{E_1} + \frac{4}{E_2}\right) \quad \text{.....(36)}$$

If we define an effective modulus, E_{eff}, by the equation

$$\frac{1}{E_{\text{eff}}} = \frac{1}{E_1} + \frac{4}{E_2} \quad \text{.........(37)}$$

Eq 36 takes the same form as Eq 1, and therefore this relaxation problem may be solved by means of Eq 33, with E_{eff} in place of E. Using $E_1 = 20 \times 10^6$ psi and assuming that $E_2 = 30 \times 10^6$ psi, Eq 37 gives $E_{\text{eff}} = 5.46 \times 10^6$ psi.

With this value of E_{eff}, Eq 33 was integrated graphically for $\sigma_0 = 10{,}000$ psi, giving the dashed curve of Fig. 8. Also shown in Fig. 8 are the data curves reproduced from Fig. 3 of Reference (**12**). In this reference, the curves are labelled "first start" and "second start" with no explanation given in the text. If it is assumed that the two curves represent different samples run in duplicate, then the two curves show the scatter of the results. On the other hand, if the curve marked "second start" represents a reloading of the first sample, then only the curve marked "first start" should be compared with the calculated curve. On the latter basis, the deviation between the observed and calculated results is uniformly about 500 to 600 psi.

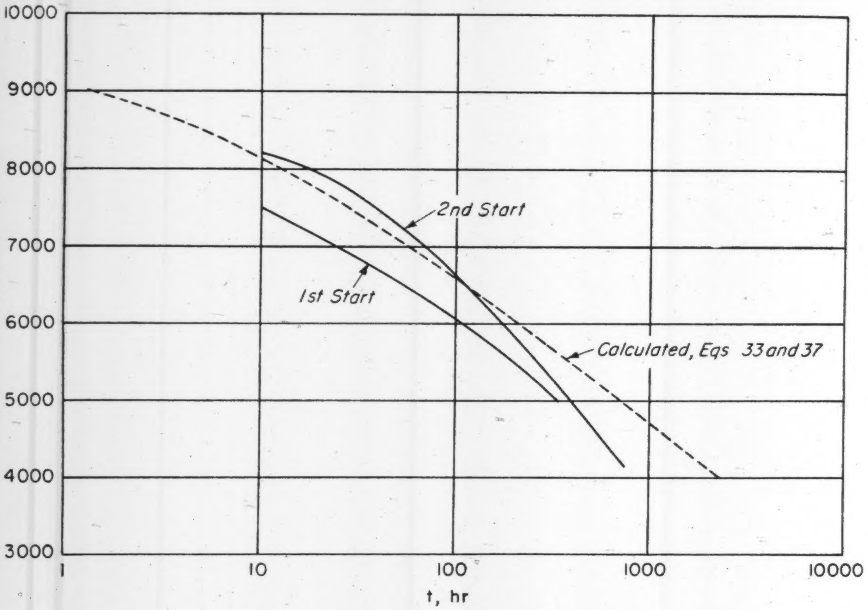

FIG. 8.—Crane "$b = 5$" Relaxation Test.
0.35 per cent carbon steel K-20 at 850 F, $\sigma_0 = 10{,}000$ psi.

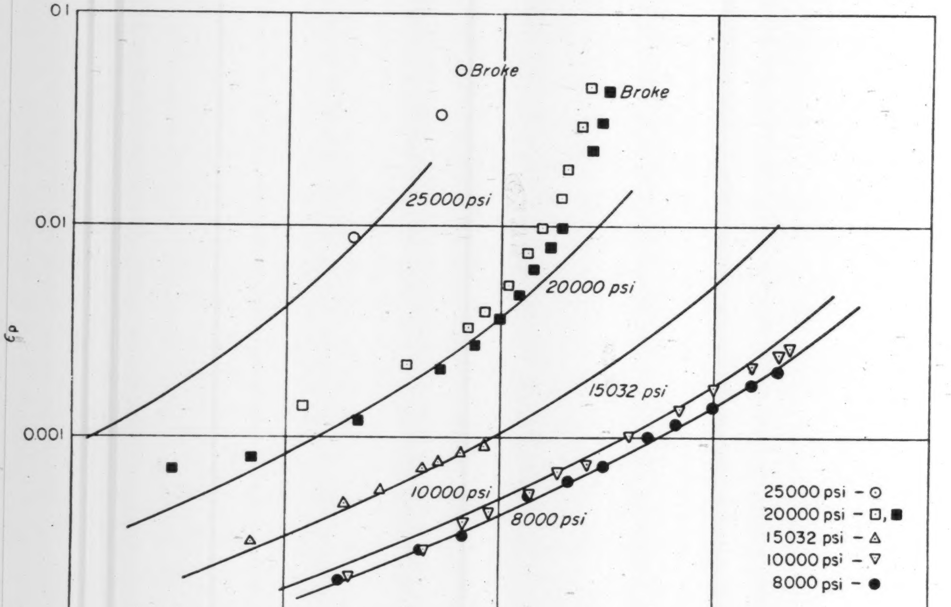

FIG. 9.—Creep of Alloy S-816 at 1500 F.
Curves calculated from Eq 38.

ALLOY S-816

Through the courtesy of Mr. H. C. Cross of the Battelle Memorial Inst., we have obtained a set of creep data and step-down relaxation data on the alloy S-816 at 1500 F. The composition and properties of this material are given by Robinson (14) as sample No. 92 of Table III. The creep data consist of two creep curves at 8000 and 10,000 psi, a short-time creep curve at 15,032 psi taken from the first step of the relaxation test, and a number of higher stress short-time rupture tests during which creep readings were taken. For most of the creep curves, the initial elastic strain was in good agreement with a modulus value of 22×10^6 psi. However, in the case of the 15,032 psi curve, the difference was significant, the observed initial elastic strain being 0.00080 as compared with a calculated value of 0.00069. It was decided to deduct the observed, rather than the calculated, initial elastic strain from the total strain in all cases, since this led to better self-consistency of the data. This is equivalent to the assumption that the initial zero setting of the extensometer was incorrect. The results are shown in Fig. 9.

TABLE II.—STEP-DOWN TEST ON ALLOY S-816 AT 1500 F.

σ, psi	$t_2 - t_1$, hr	ϵ_{p1} (observed)	$\Delta\epsilon_p$ (observed)	$\Delta\epsilon_p$ (calculated)	Deviation, $\Delta\epsilon_p$
15 032.......	86	0.00000	0.00090	0.00095	+0.00005
12 791.......	20	0.00090	0.00013	0.00007	−0.00006
10 549.......	119	0.00103	0.00018	0.00019	+0.00001
8 308.......	227	0.00121	0.00022	0.00021	−0.00001
6 066	275	0.00143	0.00006	0.00015	+0.00009

In fitting an empirical equation to the data, the greatest weight was given to the lower stress long-time creep data, rather than to the higher stress data close to the rupture point. The equation developed is

$$t = \frac{(K\epsilon_p)^n}{(1 + q\epsilon_p)^m (e^{\sigma/s} - 1)^{\sigma/s}} \quad \ldots (38)$$

in which

$K = 17{,}150$
$s = 9800$
$q = 901$
$n = 2.75$
$m = 2.00$

It is interesting to note that the creep curves of this material show a very marked displacement with stress, which is handled in Eq 38 by placing σ in the exponent of the stress function. The solid curves of Fig. 9 were calculated from Eq 38.

The results of the step-down relaxation test are given in Table II. Calculation of the increase in plastic strain occurring during each of the constant-stress periods was carried out in the same manner as for the carbon steel test described previously. Differentiation of Eq 38 to give the differential creep law by the strain-hardening assumption, followed by integration between the limits ϵ_{p_1} and ϵ_{p_2} and the limits t_1 and t_2, gives

$$\frac{\epsilon_{p_2}^n}{(1 + q\epsilon_{p_2})^m} - \frac{\epsilon_{p_1}^n}{(1 + q\epsilon_{p_1})^m} = \frac{(e^{\sigma/s} - 1)^{\sigma/s}}{K^n} (t_2 - t_1) \quad \ldots (39)$$

This equation was used to calculate the values given in the fifth column of Table II.

The agreement is good except for the last period (6066 psi) in which the deviation between the observed and calculated strain increments is 0.00009 units. Examination of the curve obtained for this period shows that the measured creep of 0.00006 units occurred during the first 100 hr, which is in agreement with the change calculated from Eq 39 for a 100-hr period. During the remaining 175 hr, no further creep was observed, indicating some erratic effect during this time.

CONCLUSION

The evidence accumulated in this paper indicates that for metals at temperatures and stresses in the range of commercial importance, the creep rate at a given temperature is determined, within the precision of the measurements, solely by the stress and by the extent of plastic strain which the material has undergone. However, this statement should not be interpreted to mean that time-hardening does not exist. Time-hardening is certainly an important and measurable effect at high temperatures, but in the range of creep rates of the experiments discussed here, it appears to be of negligible importance.

In like manner, the phenomenon of creep recovery, while it is measurable under the conditions of these experiments, appears to be of little importance. For example, examination of the step-down creep curves (10) which are the basis of Table I shows the existence of a slight amount of creep recovery immediately after reduction of stress. However, for reasonably long periods, the strain-hardening equation, Eq 34, does predict total strains which are in good agreement with experiment, although the equation cannot predict a short-time recovery. Apparently, either the amount of recovery is negligible, or subsequent creep rates are increased to make up for the recovery effect. In this connection, Phillips and Smith (6), in discussing their alternate load experiments on copper, remark "A certain degree of strain recovery is always experienced with time at low loads, but upon reloading this recovery is exactly compensated by a short initial period of accelerated extension." It is reasonable to suppose, then, that creep recovery is a completely reversible phenomenon, whose effect disappears either upon increase in stress, or after a long period at about the same stress.

Most of the statements that creep and relaxation data cannot be correlated are apparently based upon methods that require the measurement of the slopes of creep and relaxation curves in order to obtain creep rates. While the measurement of such slopes is an extremely useful means of obtaining creep or relaxation rates for industrial design purposes, it is inherently inaccurate. All creep and relaxation data show scatter because of such factors as temperature variation, imperfect rigidity of apparatus supports, and estimating in the reading of scales. To draw a smooth curve through a set of creep data requires some judgment, and to place a straight-edge tangent to the early portion of a creep curve requires considerably more judgment. Finally, in the latter portions of the creep curve which approach linearity, the strain increments usually measured are so small as to be close to the limit of precision of the measurements. These individual variations of slope measurement are eliminated by the method used in this paper, which consists of fitting a single empirical equation to all of the creep data and of differentiating this equation to give a single differential equation which expresses all of the creep rates.

It is probable that there exist in the files of other laboratories both creep and relaxation data for single materials, in which the creep data cover a wide enough range that empirical equations may be fitted with reasonable accuracy. It would be interesting to see whether the correlation of such data confirms the findings of this paper. The author has found that the labor of graphical integration of the relaxation equations is relatively small in comparison with that required for the trial and error fitting of empirical equations to the creep data. It may be possible, with sufficient data on a variety of materials over a range of temperatures, to develop standardized forms of empirical creep equations which will be suitable for the solution of relaxation problems.

Acknowledgment:

The author wishes to express his appreciation to H. C. Cross of Battelle Memorial Inst. for the data on Alloy S-816, and to C. T. Evans, Jr., and others at the Elliott Co. for their helpful discussions of these problems.

REFERENCES

(1) C. R. Soderberg, "The Interpretation of Creep Tests for Machine Design," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 58, pp. 733–743 (1936).

(2) E. P. Popov, "Correlation of Tension Creep Tests with Relaxation Tests," *Transactions*, Am. Soc. Mechanical Engrs., *Journal of Applied Mechanics*, Vol. 69, pp. A-135 (1947).

(3) E. A. Davis, "Creep and Relaxation of Oxygen-Free Copper," *Transactions*, Am. Soc. Mechanical Engrs., *Journal of Applied Mechanics*, Vol. 65, p. A-101 (1943).

(4) I. Roberts, "Correlation of Tension Creep Tests with Relaxation Tests," *Transactions*, Am. Soc. Mechanical Engrs., *Journal of Applied Mechanics*, Vol. 71, p. 208 (1949).

(5) C. C. Davenport, Discussion on "The Interpretation of Creep Tests for Machine Design," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 59, pp. 511–512 (1937).

(6) A. J. Phillips and A. A. Smith, Jr., "Effect of Time on Tensile Properties of Hard-Drawn Copper Wire," *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part II, pp. 263–273 (1936).

(7) C. C. Davenport, "Correlation of Creep and Relaxation Properties of Copper," *Transactions*, Am. Soc. Mechanical Engrs., *Journal of Applied Mechanics*, Vol. 60 p. A-55 (1938).

(8) H. C. Cross and F. B. Dahle, "Long-Time Creep Tests of 18Cr-8Ni Steel and 0.35 per cent Carbon Steel," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 58, pp. 91–96 (1936).

(9) H. C. Cross and J. G. Lowther, "Long-Time Creep Tests of 18Cr-8Ni Steel K-19 and 0.35 C Steel K-20," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 59, pp. 441–445 (1937).

(10) E. L. Robinson, "A Relaxation Test on 0.35 C Steel K20," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 59, pp. 451–452 (1931).

(11) "Compilation of Available High-Temperature Creep Characteristics of Metals and Alloys," published jointly by the Am. Soc. Testing Mats. and the Am. Soc. Mechanical Engrs., March, 1938.

(12) E. L. Robinson, "The Resistance to Relaxation of Materials at High Temperature," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 61, pp. 543–550 (1939).

(13) *Ibid*, discussion by John Boyd, pp. 551–552.

(14) E. L. Robinson, "High Temperature Bolting Materials," *Proceedings*, Am. Soc Testing Mats., Vol. 48, pp. 214–235 (1948).

DISCUSSION

MR. EVAN A. DAVIS[1] (*presented in written form*).—The author of this paper has shown such good agreement between his derived relaxation curves and the experimentally obtained ones that the casual reader might reach the conclusion that relaxation tests are no longer necessary. This discussion was prepared to discourage the formation of such conclusions and to comment on some factors which have been shown by experiment to affect the behavior of material.

In his correlation of creep and relaxation data, the author has treated the problem as though the behavior of material at a given temperature depends only upon the stress, the plastic strain, and the plastic strain rate. In other words, he assumes that the only factors that need to be considered are "speed-effect" and "strain hardening effect." I agree that these are the two most important factors, but I am not willing to admit that they are the only factors to be considered. In some materials aging or annealing are important factors, and it has been demonstrated that recovery definitely affects the latter stages of a relaxation test.

Two of the materials used for correlation purposes, the slightly cold-worked copper at 165 C and the K-20 steel at 850 F, were of a very stable nature, and it is not too surprising that the lack of consideration of effects depending upon time alone did not cause wide disagreement between predicted and observed results. The amount of prestretching of the copper was not great enough to cause any appreciable amount of annealing at 165 C. The K-20 steel was chosen originally because of its uniformity and stability, and it was tested in a fully annealed state.

The effect of recovery during a relaxation test has been overshadowed in the author's correlation of data by his adoption of a constantly diminishing creep rate in the creep test as is shown in his Eqs 23 and 32. The effect of recovery is a slowing down in the rate of relaxation. The effect of a continuously diminishing creep rate at constant stress is also a slowing down of the rate of relaxation. When one effect is substituted for the other there is a certain amount of compensation, but it is very unlikely that these two effects will balance each other exactly. It has been shown[2] that in a long-time relaxation test the stress may fall at a lower rate than that predicted by a theory which takes into account the speed effect and the maximum amount of strain hardening effect consistent with the existence of a minimum creep rate.

If the factors and effects just described are properly considered it seems, that the methods of prediction developed in the paper may be applied quite well to materials that are thermally and metallurgically stable, but that they should be used with caution for all other materials.

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[1] Advisory Engineer, Westinghouse Electric Corp., East Pittsburgh, Pa.

[2] Evan A. Davis, Creep and Relaxation of Oxygen Free Copper, *Journal of Applied Mechanics*, pp. A100–A105 June, 1943.

MR. YOH-HAN PAO[3] (*by letter*).—Mr. Roberts is to be complimented on a clear and concise study of the creep of metals under varying uniaxial stress and the present paper should indeed help to clear up some of the confusion that has existed in this field.

In particular, Fig. 1 and Eq 4, 5, and 6 are very helpful in pointing out that an empirical relation obtained under conditions of constant stress should not be applied indiscriminately to conditions of varying stress.

It is to be regretted, however, that Mr. Roberts has limited himself to a discussion of cases where all the creep strain is permanent and there is no creep recovery whatever.

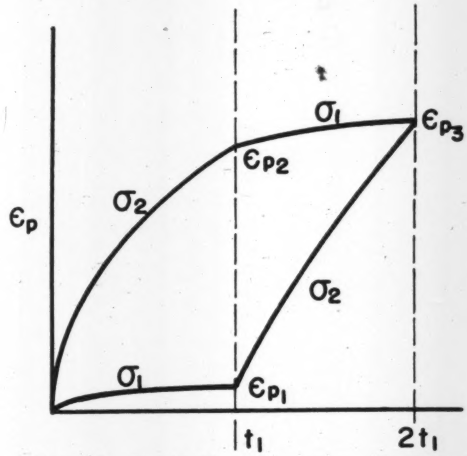

FIG. 10.—Strain Hardening Law. No Creep Recovery. ϵ_{p3} Independent of Path of Loading.

For example, Fig. 2 represents an alternate load experiment. It was found that the total creep strain was independent of the order of applying the loads. This may not be true in the general case where some of the creep strain is recoverable. This point is illustrated in the Figs. 10 and 11. For the sake of making the point clear, the loads are assumed to be 2 per cent and 70 per cent of the tensile strength.

Figure 12 shows two constant-stress creep curves obtained for a material at constant temperature and at stress levels σ_1 and σ_2.

Now, suppose a specimen is subjected to σ_1 for a period of time t_1 so that the specimen reaches the point *A* and the creep strain is ϵ_{p_1}. Then, the stress is suddenly decreased to σ_2. According to the different assumptions, this is what would happen in each case.

1. *Strain-Hardening Law.*—The load is assumed to be decreased instantaneously so that no recovery takes place during the unloading. No recovery is assumed to take place after this either. Right after unloading, the specimen is assumed to have the creep rate of point *A′* and if the specimen is then held at σ_2 for a period of time t_2, the whole creep curve would be *OAB* where *AB* is of the same shape as *A′B′*. This interpretation is based on Davenport's work,[4, 5] Popov's work[6] and the author's interpretation implied by Fig. 2.

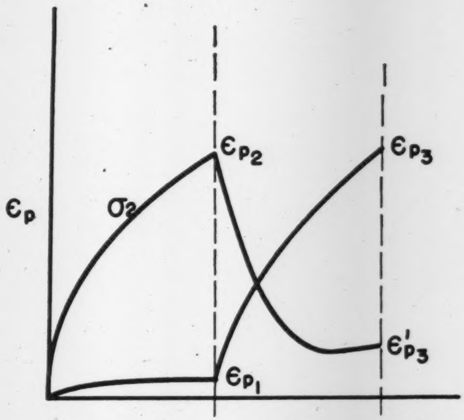

FIG. 11.—Some Creep Recovery Takes Place in General.

$\epsilon_{p3} \neq \epsilon_{p'3}$

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[3] Research Associate, Dept. of Engineering Mechanics, The Pennsylvania State College, State College, Pa.

[4] C. C. Davenport, "Correlation of Creep and Relaxation Properties of Copper," *Journal of Applied Mechanics*, Vol. 5, No. 2, July, 1938.

[5] C. C. Davenport, Discussion on "The Interpretation of Creep Tests for Machine Design," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 59, pp. 511–512 (1937).

[6] E. P. Popov, "Correlation of Tension Creep Tests with Relaxation Tests," *Journal of Applied Mechanics*, Am. Soc. Mechanical Engrs., Vol. 69, p. A-135 (1947).

2. *Time-Hardening Law.*—Again no recovery is assumed to take place during or after unloading. Right after the load is decreased to σ_2, the creep rate is the same as that of point C and if the specimen is then held at σ_2 for a period of time t_2, the whole creep curve would be OAD where AD is the same as CD'.

If all of the creep strain is recoverable and if the recovery happens instantaneously, then the creep strain right after unloading is that of point C and the whole creep curve would be $OACD'$.

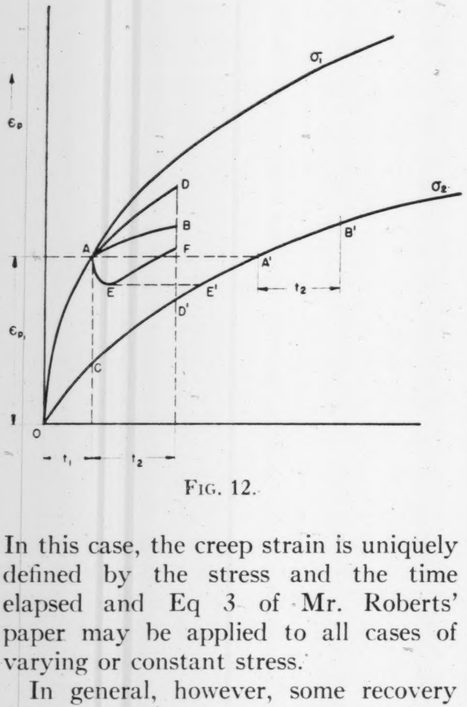

FIG. 12.

In this case, the creep strain is uniquely defined by the stress and the time elapsed and Eq 3 of Mr. Roberts' paper may be applied to all cases of varying or constant stress.

In general, however, some recovery will take place, but the action of recovery will take some time. The creep curve will be $OAEF$.

The behavior of the material under these conditions would seem therefore to be rather complicated, and neither the strain-hardening law nor the time-hardening law can take care of the recovery (AEF in Fig. 12). The strain-hardening law is also often expressed mathematically as

$$u \ \mathrm{f}(\epsilon_p, \sigma)$$

where:

u = the creep rate at any time, t,

ϵ_p = the creep strain at that time, t, and

σ = stress at any time, t.

As formulated this way, the expression $u = \mathrm{f}(\epsilon_p, \sigma)$ is identical to the strain-hardening law and cannot take care of creep recovery either.

However, if this relationship between the creep rate, creep strain, and applied stress is somewhat modified and applied to the creep "components" separately, the simultaneous action of creep and creep recovery is brought out very clearly, and the behavior of the material under the action of varying uniaxial stress can be described and predicted more accurately.

This is brought out in the following analysis.

Let us consider a hypothetical material which has the following characteristics:

The total strain of this material under sustained constant tensile load may be considered to be made up of three components. The components are:

(*a*) The elastic strain—which is proportional to the applied load.

(*b*) The recoverable creep strain—this is often called the transient creep strain. It is assumed that it remains constant with time after it reaches a maximum value. It is entirely recoverable and the recovery curve is the reverse of the creep curve.

(*c*) The permanent creep strain.

Figure 13 represents the creep curve of such a material and also shows how it may be considered to be made up of the three components.

Thus, creep strain $= \epsilon_p = \epsilon'_p + \epsilon''_p = K\sigma^{\alpha}(1 - e^{-pt}) + B\sigma'' t$.

Figure 14 shows the full recovery curve of such a material. The elastic strain is not shown.

Much evidence exists to show that many actual materials have creep characteristics similar to those shown in these two figures. Furthermore, it has been shown[7] that these two kinds of creep strains may be due to different physical phenomena and indeed may obey different physical laws. The one may be predominant at low and moderate temperatures, whereas the other is predominant at high temperatures. The results derived further on are, therefore, of more than mere theoretical interest.

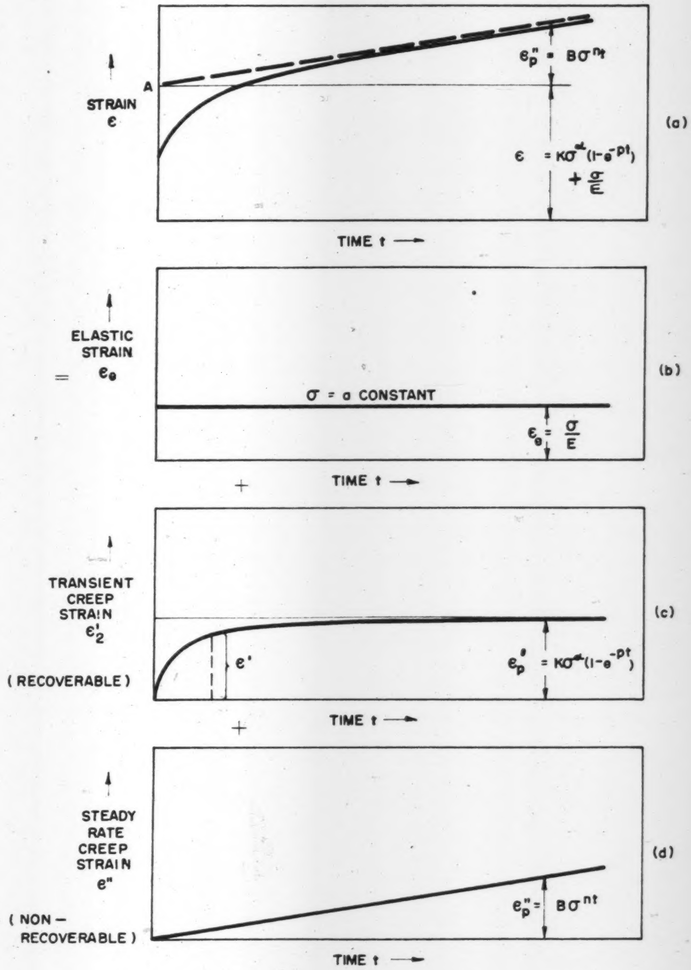

FIG. 13.—Components of Creep Strain.

Now, let us assume that the recoverable component obeys the relation $u' = f(\epsilon'_p, \sigma)$. Let us say that the recoverable portion of the creep curve under constant load can be approximated by

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[7] E. N. Andrade, "On the Viscous Flow in Metals and Allied Phenomena," *Proceedings*, The Royal Soc., London, Series A, Vol. 84 (1910–1911).

$$\epsilon_p' = K\sigma^{\alpha}(1 - e^{-pt}) \ldots\ldots\ldots (1)$$

Then

$$u' = pK\sigma^{\alpha}e^{-pt} \ldots\ldots\ldots\ldots (2)$$

or, in general

$$u' = p(K\sigma^{\alpha} - \epsilon_p') \ldots\ldots\ldots\ldots (3)$$

Equation 3 is of the form $u' = f(\epsilon_p', \sigma)$.

Thus, in Fig. 14, Eq 3 at first represents the conditions along the curve oA', but as soon as the load is removed, the stress is zero but the transient creep strain is still that of point A'; therefore, substituting these values in Eq 3 we have

$$u_{A'} = -p\epsilon_p' \ldots\ldots\ldots\ldots (4)$$

Integration of this within the proper limits will give an exponential curve which is geometrically similar to oA' but is in an upside down position. Thus, Eq 4 predicts the negative creep strains and represents the curve AB.

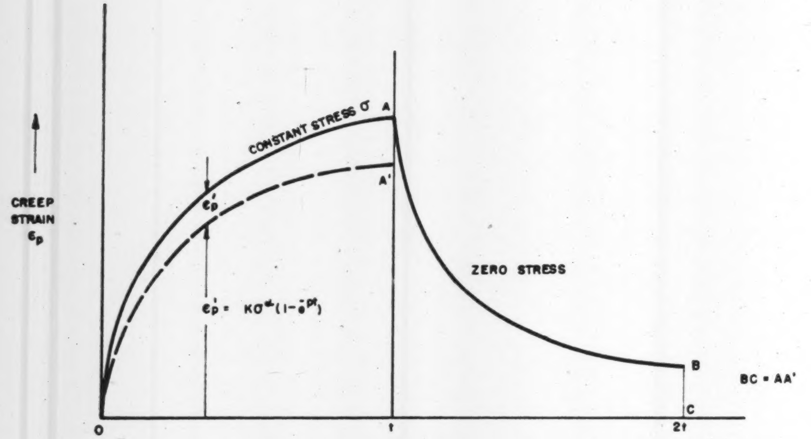

FIG. 14.—Creep Recovery.

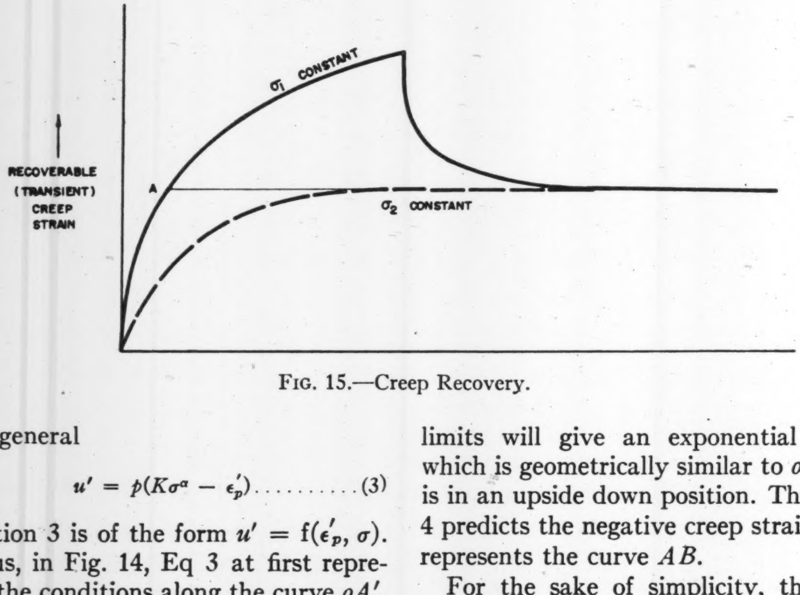

FIG. 15.—Creep Recovery.

For the sake of simplicity, the case shown in Fig. 14 is that of full recovery, but the same would be true if the stress were lowered to σ_2 instead of being completely removed. For example, the negative rate after unloading would be

$$u' = p(K\sigma_2^{\alpha} - \epsilon_p') \ldots\ldots\ldots (5)$$

This is shown in Fig. 15. It should be noted that in Eq 5 at first $K\sigma_2^{\alpha} < \epsilon'_p$. When ϵ'_p becomes equal to $K\sigma_2^{\alpha}$, no further recovery takes place.

So far, we have only discussed the transient creep strain. Actually, for the material we have specified, both the recoverable and non-recoverable components are present.

It is assumed that the non-recoverable component obeys the strain-hardening law or the relation $u'' = \mathrm{f}(\epsilon''_p, \sigma)$ where no creep recovery takes place. It is assumed further that this creep rule is approximately only a function of stress alone such that $u'' = B\sigma''$. Then, under constant load conditions, the creep strain is

$$\epsilon_p = \epsilon_p' + \epsilon'' = K\sigma^{\alpha}(1 - e^{-pt}) + B\sigma^{n}t \ldots (6)$$

The creep rate at any point is given by

$$u = u' + u_p'' = p(k\sigma^{\alpha} - \epsilon_p') + B\sigma^{n} \ldots (7)$$

Now referring to Fig. 12, we see that just before unloading, the creep rate of the material at A is

$$u_A = p(K\sigma_1^{\alpha} - \epsilon_{pA}') + B\sigma_1^{n}$$

Right after unloading, the creep rate is

$$u_A = p(K\sigma_2^{\alpha} - \epsilon_{pA}') + B\sigma_2^{n} \ldots\ldots (8)$$

The term $p(K\sigma_2^{\alpha} - \epsilon'_p)$ may represent the negative creep rate or the recovery rate. It decreases in magnitude as the recoverable creep strain is recovered. At point E the negative creep rate is equal to the positive creep rate of the non-recoverable component, and so the net creep rate is zero at this point.

For actual materials, all the experimental constant needed for this method of analysis could be obtained from creep and creep recovery tests.

In view of the good results obtained from the correlation of relaxation and creep data of metals by means of the strain hardening law, it may seem at first thought that this more detailed analysis may not be justified or worth while. However, it should be noted that this method is a more general one, and in the case where creep recovery is negligible, it becomes identical to the strain hardening law. Figure 15 also shows that there is no creep recovery when the stress is decreased from σ_1 to σ_2 if the specimen has not progressed beyond point A during the time it was stressed at σ_1. This may happen in the relaxation of metals, especially if the total length of time of test is relatively short.

This analysis by components is therefore useful in the treatment of creep and relaxation of metals and plastics in cases where the creep recovery is appreciable but at the same time is general enough so that if creep recovery is negligible it is still valid.

MR. IRVING ROBERTS (*author's closure*).—The author is in general agreement with Mr. Davis' cautioning remarks with regard to possible time-hardening and recovery effects in relatively unstable materials. Until considerably more data are available on the creep and relaxation of a variety of materials, it is not certain that the correlation will be of general application. However, up to the present time, this author has not been able to find any set of creep and relaxation data which would tend to demonstrate that the accuracy of the correlation is poor for any material.

Mr. Pao presents one possible method of attack on the creep recovery problem, that of dividing the creep into recoverable and nonrecoverable portions with arbitrarily assumed creep equations. This method may prove valuable when a considerable amount of creep recovery data becomes available for correlation.

MECHANICAL PROPERTIES IN TORSION AND POISSON'S RATIO FOR CERTAIN STAINLESS STEEL ALLOYS*

By C. W. Muhlenbruch,[1] V. N. Krivobok,[2] and C. R. Mayne[2]

Synopsis

Modulus of elasticity in shear, shearing yield strength, and Poisson's ratio have been studied for a number of AISI 300 series stainless steels. The torque-twist relationships are non-linear as are the tensile or compressive stress-strain diagrams. Values for secant shearing modulus of elasticity and a yield strength at a limiting amount of torsional strain are given as applicable to design problems. Work hardening reduces the shearing modulus and increases the shearing yield strength. Poisson's ratio for longitudinal specimens is almost constant with increasing stress up to, and in some cases beyond, the yield strength. The ratio increases with increasing stress for transverse specimens. Harder tempers have higher Poisson's ratios than softer tempers. Reasons for these fundamental behaviors are advanced, based on an analysis of the stress-strain relationships, the susceptibility to work hardening, and the state of stress in the material when subjected to axial loading. The usual value of Poisson's ratio of 0.30 may be satisfactory in some cases but is in considerable error in others. A theoretical relationship between Poisson's ratio and secant moduli in tension and shear is presented.

Designers employing stainless steel for structural components in aircraft and railroad cars have long had need for information on the torsional properties of this material. There has also been uncertainty as to the constancy of Poisson's ratio with increasing stress when the stress-strain relationship is non-linear and the material is anisotropic. Recent developments in jet aircraft and guided missiles have accentuated the need for these properties. The work reported in this paper presents such information in a fundamental way for the more important stainless steel structural alloys. Certain fundamental modes of behavior, based on the susceptibility of various alloys to respond to cold-working, are given as axiomatic and necessary prerequisites to enlarging the scope of the research work.

Object of Investigation:

The object of this investigation was three-fold as follows for a number of AISI type stainless steel alloys of various tempers and representative of materials in common use:

1. To determine the modulus of elasticity in shear.
2. To determine Poisson's ratio in the transverse and longitudinal directions as a function of stress.
3. To correlate shearing modulus of elasticity and Poisson's ratio.

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Alloys and Test Specimens:

Standard AISI stainless steel alloys were used in this investigation. These included types 301, 302, 304, 316, 321, and 347 in the form of sheet and tubing in annealed and cold-worked tempers, although not each form and temper were tested for each alloy.

Table I identifies the specimen series designation, the alloy number, temper, chemical analysis, method of cold working, and form of the material. The mechanical properties of the materials are given in Table II. These values were obtained from data supplied by the producer or from actual physical tests. Torsion specimens are identified by a "T" followed by the alloy type number, the specimen number, the symbol "t" for tubing and finally the temper. For example, specimen T302-2t-1/2 is specimen 2 of type 302 one-half hard tubing. The symbol t was used since it is contemplated that future investigations will include some rod specimens. Poisson's ratio specimens were identified in the same way but with the prefix P and with p and t to indicate directions parallel and transverse to rolling. Thus P301-1p-1/2 indicates Poisson's ratio specimen of type 301 one-half hard sheet cut parallel to the direction of rolling.

TABLE I.—IDENTIFICATION OF STAINLESS STEEL SAMPLES TESTED.

Specimen Series Designation	AISI Alloy Designation	Temper	Analysis, per cent									Material Form
			Carbon	Manganese	Phosphorus	Sulfur	Silicon	Copper	Nickel	Chromium	Other	
T301-t-1/4	301	1/4 Hard	0.092	...	...	...	...	...	7.65	17.93	...	Tubing
T301-t-1/2		1/2 Hard	0.092	...	...	...	...	...	7.65	17.93	...	Tubing
P301-An		Annealed	0.111	1.35	...	...	0.53	...	7.45	17.66	...	Sheet
P301-1/2		1/2 Hard	0.10	1.41	0.32	0.011	0.42	...	7.01	17.54	...	Sheet
P301-Full		Full Hard	0.09	1.48	0.027	0.015	0.48	0.08	7.28	17.56	...	Sheet
T302-t-1/2	302	1/2 Hard	0.10	0.88	0.019	0.012	0.46	...	11.19	18.34	...	Tubing
P302-An		Annealed	0.113	1.12	...	...	0.63	...	9.95	17.88	...	Sheet
T304-t-An	304	Annealed	0.05	0.43	...	...	0.25	...	10.56	18.12	...	Tubing
T304-t-1/4		1/4 Hard	0.06	1.73	...	...	0.41	...	10.18	18.44	...	Tubing
T304-t-1/2		1/2 Hard	0.068	1.68	...	...	0.37	...	10.36	18.89	...	Tubing
P304-An		Annealed	0.072	1.17	...	...	0.74	...	9.87	18.52	...	Sheet
P304-1/4		1/4 Hard	0.06	1.21	0.024	0.006	0.41	0.08	8.85	18.64	...	Sheet
P304-1/2		1/2 Hard	0.07	1.15	0.025	0.023	0.35	0.14	8.76	18.70	...	Sheet
T316-t-1/4	316	1/4 Hard	0.06	1.43	...	...	0.38	...	13.10	16.71	2.31 Mo	Tubing
T316-t-1/2		1/2 Hard	0.06	1.69	...	...	0.25	...	13.00	16.63	2.45 Mo	Tubing
T321-t-1/4	321	1/4 Hard	0.073	1.50	0.021	0.012	0.55	...	11.24	18.19	0.55 Ti	Tubing
T321-t-1/2		1/2 Hard	0.073	1.50	0.021	0.012	0.55	...	11.24	18.19	0.55 Ti	Tubing
P321-1/2		1/2 Hard	0.06	1.50	0.028	0.008	0.39	0.12	9.91	17.38	0.47 Ti	Sheet
T347-t-An	347	Annealed	0.04	1.42	...	...	0.58	...	11.04	17.60	0.58 Cb	Tubing
T347-t-1/4		1/4 Hard	0.07	1.52	...	...	0.43	...	12.69	18.69	0.87 Cb	Tubing
T347-t-1/2		1/2 Hard	0.072	1.74	...	...	0.41	...	12.20	17.71	0.91 Cb	Tubing
P347-An		Annealed	0.058	1.28	...	...	0.45	...	10.64	18.01	0.78 Cb	Sheet
P347-1/4		1/4 Hard	0.04	1.25	0.15	0.006	0.42	0.30	10.95	17.60	0.53 Cb	Sheet
P347-1/2		1/2 Hard	0.06	1.37	...	...	0.59	...	10.62	18.51	0.63 Cb	Sheet

Three duplicate test specimens were tested for torsion and two for Poisson's ratio. The tubing specimens used for the torsion test received no special preparation other than cutting to a standard length of 24 in.

Details of the test specimens for Poisson's ratio are given in Fig. 1. In the first part of the investigation specimens Type 1 and Type 2 were checked for axial loading by comparing distribution of strain across the face. A greater difference in strain from one edge to the other was observed for the wide specimen, Type 1, and distribution was not always linear as for the narrower specimen, Type 2. This comparison was obtained for one specimen of each type of 301 one-half hard and 347 annealed. Data reported in this paper for these alloys is identified for this variation in specimen width. Inasmuch as the strain distribution was linear across the face of the Type 2 specimen, it was decided to make the remaining tests with this narrower type but using the gage arrangement shown for Type 3 in Fig. 1. The A-12 type of SR-4 electric strain gage was selected because it is narrow, permitting it to be mounted very close to the edge of the specimen. The type A-5 gage was selected because it is short and fits nicely across the transverse dimension of the specimen. In working up the data, the transverse gage readings were corrected for the Poisson effect. The gage

TABLE II.—TENSILE MECHANICAL PROPERTIES OF STAINLESS STEEL SAMPLES. DATA FURNISHED BY PRODUCERS.

Specimen Series Designation	Material Form	Yield Strength, psi	Tensile Strength, psi	Elongation in 2 in., per cent	OD and ID or Thickness, *t*, in.
T301-t-1/4	Tubing	84 337	130 680	37.5	OD = 0.752 ID = 0.645
T301-t-1/2	Tubing	117 492	169 330	20.3	OD = 0.880 ID = 0.780
P301-An	Sheet	34 000	102 000	72.0	*t* = 0.126
P301-1/2	Sheet	106 700	150 150	24.5	*t* = 0.076
P301-Full	Sheet	139 400	189 500	10.0	*t* = 0.093
T302-t-1/2	Tubing	...	152 000	14.0	OD = 0.878 ID = 0.774
P302-An	Sheet	37 500	90 000	58.0	*t* = 0.103
P302-1/2	Sheet	137 550	171 600	9.0	*t* = 0.079
T304-t-An	Tubing	38 000	76 100	73.0	OD = 0.842 ID = 0.600
T304-t-1/4	Tubing	87 300	110 000	41.0	OD = 1.002 ID = 0.875
T304-t-1/2	Tubing	127 000	169 750	13.0	OD = 1.054 ID = 0.813
P304-An	Sheet	38 500	87 500	66.0	*t* = 0.128
P304-1/4	Sheet	103 900	131 250	17.5	*t* = 0.063
P304-1/2	Sheet	136 700	172 400	9.0	*t* = 0.059
T316-t-1/4	Tubing	111 100	126 250	19.0	OD = 0.996 ID = 0.875
T316-t-1/2	Tubing	110 300	167 000	12.0	OD = 1.057 ID = 0.814
T321-t-1/4	Tubing	...	115 000	24.0	OD = 0.503 ID = 0.406
T321-t-1/2	Tubing	...	131 000	14.0	OD = 0.502 ID = 0.402
P321-1/2	Sheet	132 100	156 600	7.0	*t* = 0.093
T347-t-An	Tubing	50 140	93 960	43.0	OD = 0.839 ID = 0.636
T347-t-1/4	Tubing	80 000	113 000	23.5	OD = 1.004 ID = 0.870
T347-t-1/2	Tubing	136 000	158 000	13.5	OD = 1.050 ID = 0.817
P347-An	Sheet	41 500	88 500	53.5	*t* = 0.120
P347-1/4	Sheet	120 100	141 600	19.0	*t* = 0.064
P347-1/2	Sheet	130 000	162 000	7.0	*t* = 0.076

factor supplied by the manufacturer provided a satisfactory correction for the longitudinal readings. It will be noted from Fig. 1 that a total of 4 gages were mounted parallel to the longitudinal direction of the specimen and 2 in the transverse direction. The use of companion gages in this fashion permitted a satisfactory average determination of longitudinal and transverse strain. The gages were carefully positioned so that they were opposite one another and aligned in the longitudinal and transverse directions. As shown in Fig. 1, the test specimens were long enough so that sufficient material was available between grips to prevent any irregularities in surface strains due to bending of the specimen. The shank ends of the specimens were held in Templin-type grips mounted on spherically-seated tension bolts further to insure alignment of load. The width of the specimen was such that it could be fitted into these grips without binding due to contact with the sides of the grip cavity.

Electric strain gages were applied with Duco household cement after the surface was cleaned with carbon tetrachloride and acetone and precoated in the prescribed manner. Specimens were allowed to dry 24 hr prior to testing. As a further precaution, all gages were coated with beeswax after the glue had dried to prevent moisture change of the backing paper.

Testing Equipment and Test Procedure:

The tests for modulus of elasticity in shear were carried out in an Olsen 50,000 in-lb. torsion machine. This apparatus was fitted with self-aligning gripping devices to permit uniform application of torsional load without bending. Load was applied by hand in the range in which torque-twist data was obtained and with an electric motor to failure. Torsional strain, in degrees, was obtained with an Olsen-Muhlenbruch troptometer set for a 10-in. gage length. This instrument reads directly to 0.2 deg and by estimation to 0.1 deg.

Steel plugs were used to prevent the tubing from buckling. One plug was inserted from each end of the specimen. Since two plugs were used, they offered

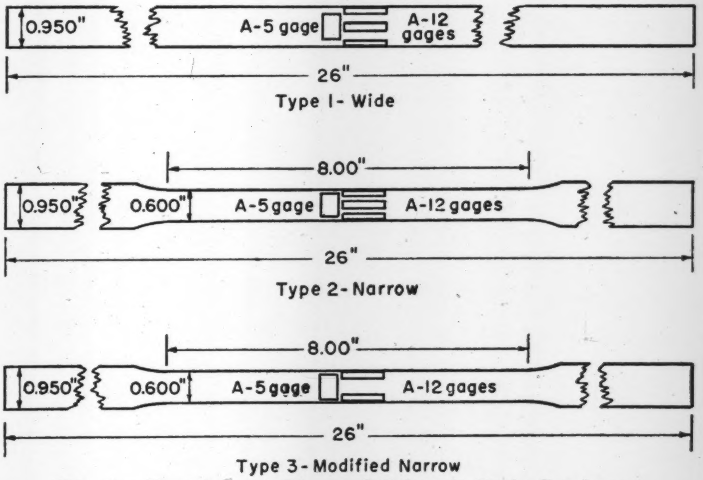

FIG. 1.—Experimental Types of Poisson's Ratio Specimens.

Type 3 was finally selected for almost all of the specimens. The gage pattern on the back face is a duplicate of this side.

no restraint and therefore did not effect the angle of twist or the torque. The plugs were made to a snug fit in the gripped end of the specimen but were approximately 0.015 in. smaller in diameter in the center portion. This permitted reduction in diameter of the tubing under test and also facilitated removal of the plugs after the test was completed.

A set procedure was followed in testing all specimens. The troptometer was attached to the tube with the plugs in place, and the specimen was gripped in the jaws of the testing machine. The troptometer was adjusted to zero at no torque, and readings of torsional strain were taken at equal increments of torque until the specimen was well into the plastic range. Increments of torque used for the different sizes of specimen are apparent from the torque-twist diagrams. When severe plastic deformation had been observed, the troptometer was disconnected, and the specimen was loaded to failure.

Poisson's ratio tests were made in a 60,000-lb Baldwin-Southwark hydraulic testing machine using a 6000 or 24,000-lb scale range. Load was applied in such magnitudes that even increments of stress were produced. The first stress in most cases was 2000 psi and was considered the initial load. It was applied to straighten and align the specimen and grips in the testing machine. Strain differences were obtained with respect to this initial reading.

Strains were measured with a Baldwin SR-4 strain indicator which permits reading to 0.000001 in. per in. A multiple

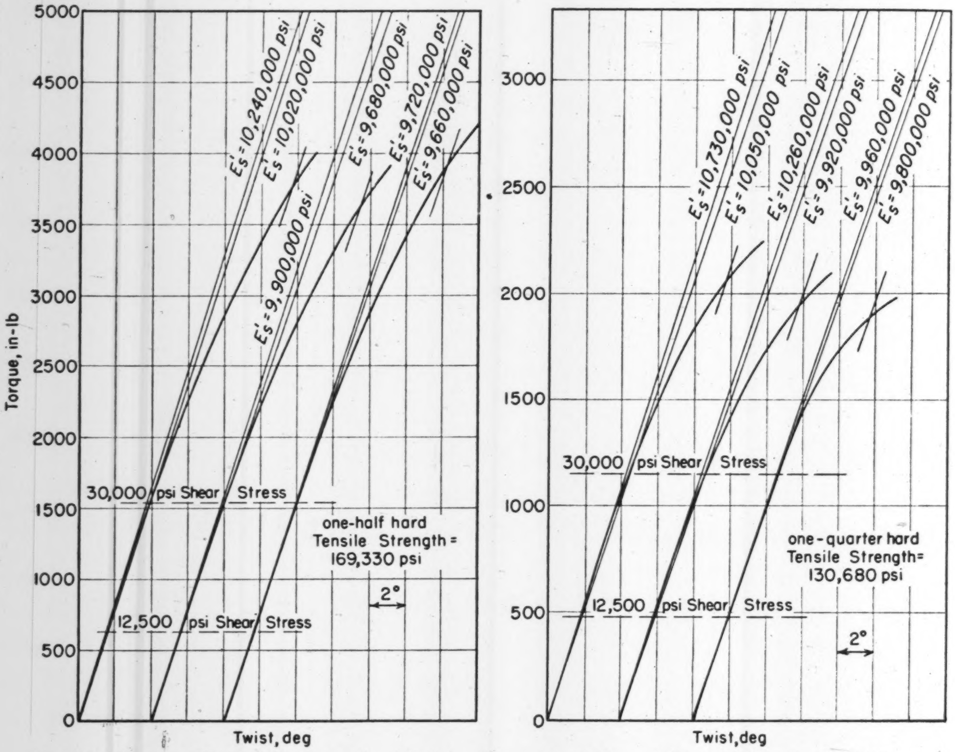

FIG. 2.—Torque-Twist Diagrams for Various Tempers of Type 301 Stainless Steel Tubing.

mercury switching device was used to permit rapid switching from one gage to another, making it possible to read all gages within a maximum of 2 min after the load has been reached. Two men were used to run each Poisson's ratio test, one to apply and hold the load and the other to read and record the data.

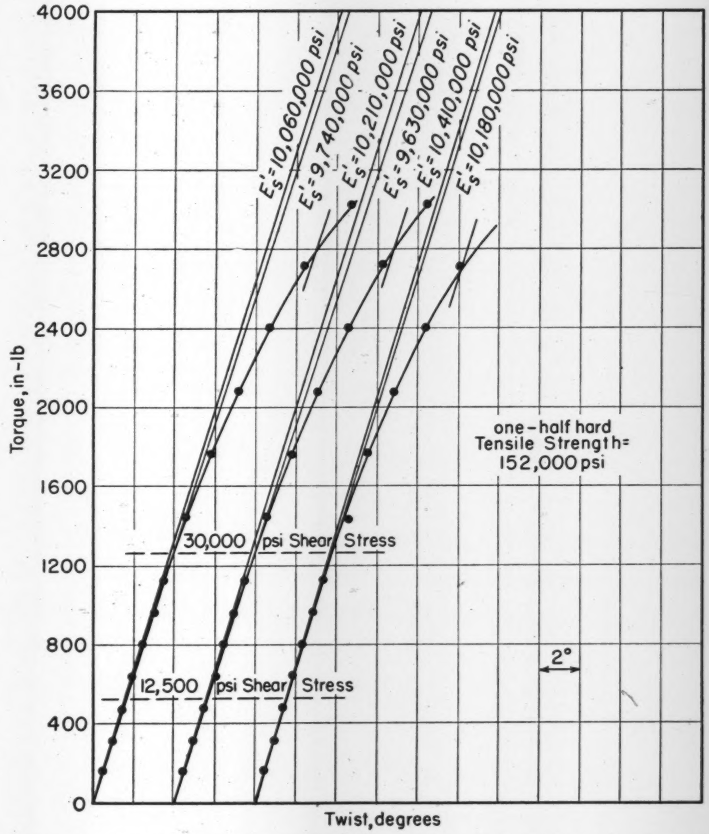

FIG. 3.—Torque-Twist Diagrams for 3 Specimens of Type 302 One-half Hard Stainless Steel Tubing.

Data points have been shown for these specimens as typical of the uniformity of the test results. The data points have not been shown in the other figures because of the scale of reproduction.

RESULTS OF TESTS

Torque-Twist Diagrams:

Figures 2 through 7 are torque-twist diagrams for the "elastic" and beginning "plastic" range. A typical set of data points are shown in Fig. 3 for type 302 one-half hard stainless steel. Actual data points have not been given on the other plots due to the small scale of reproduction. In all cases the data points were very regular and closely comparable. Torque-twist diagrams for companion specimens are considered to be in good agreement.

It should be noted that specimens of different alloys and tempers varied in size so that the diagrams are not directly comparable. Any value of torque can be converted to shearing stress in the outer

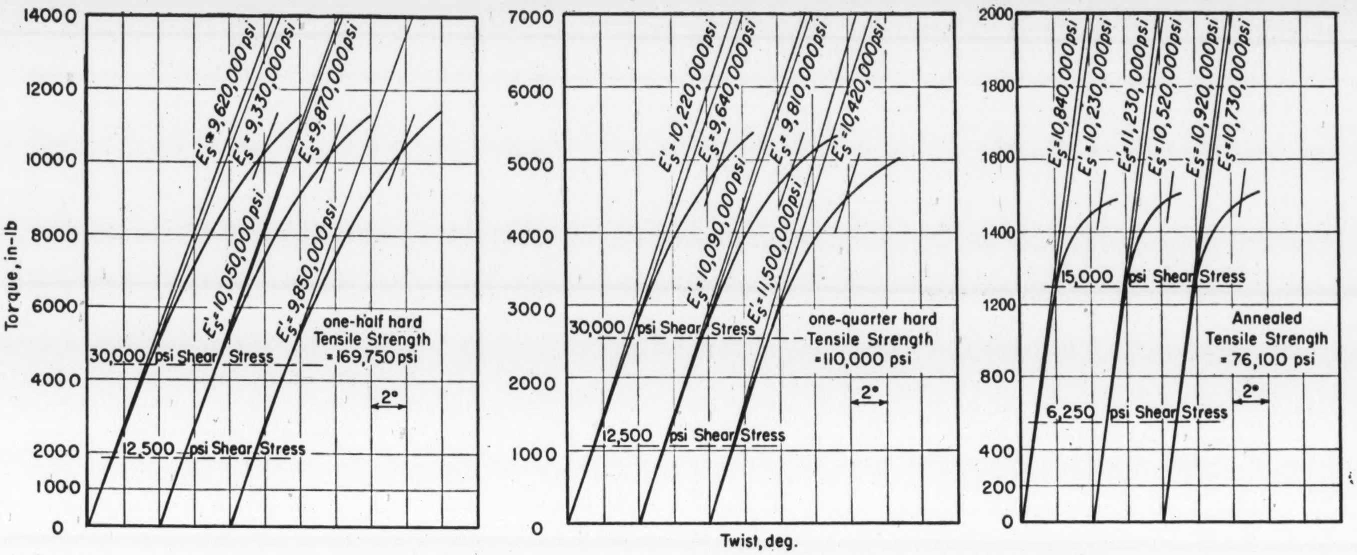

FIG. 4.—Torque-Twist Diagrams for Various Tempers of Type 304 Stainless Steel Tubing.

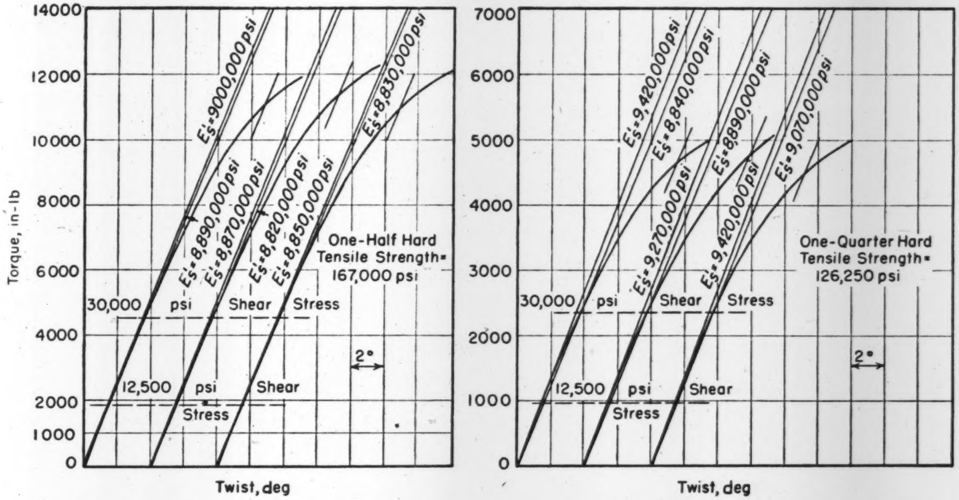

FIG. 5.—Torque-Twist Diagrams for Various Tempers of Type 316 Stainless Steel Tubing.

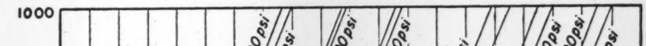

FIG. 6.—Torque-Twist Diagrams for Various Tempers of Type 321 Stainless Steel Tubing.

fiber of the tube from the following formula developed from strength of materials:[3]

$$s_s = T\frac{5.1d_0}{d_0^4 - d_i^4}$$

where:

s_s = maximum shearing stress in tube for the torque, T, psi,
T = torque, in-lb,
d_0 = outside diameter of tube, in., and
d_i = inside diameter of tube, in.

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[3] Joseph Marin, "Strength of Materials," p. 71, Macmillan Co., First Edition (1949).

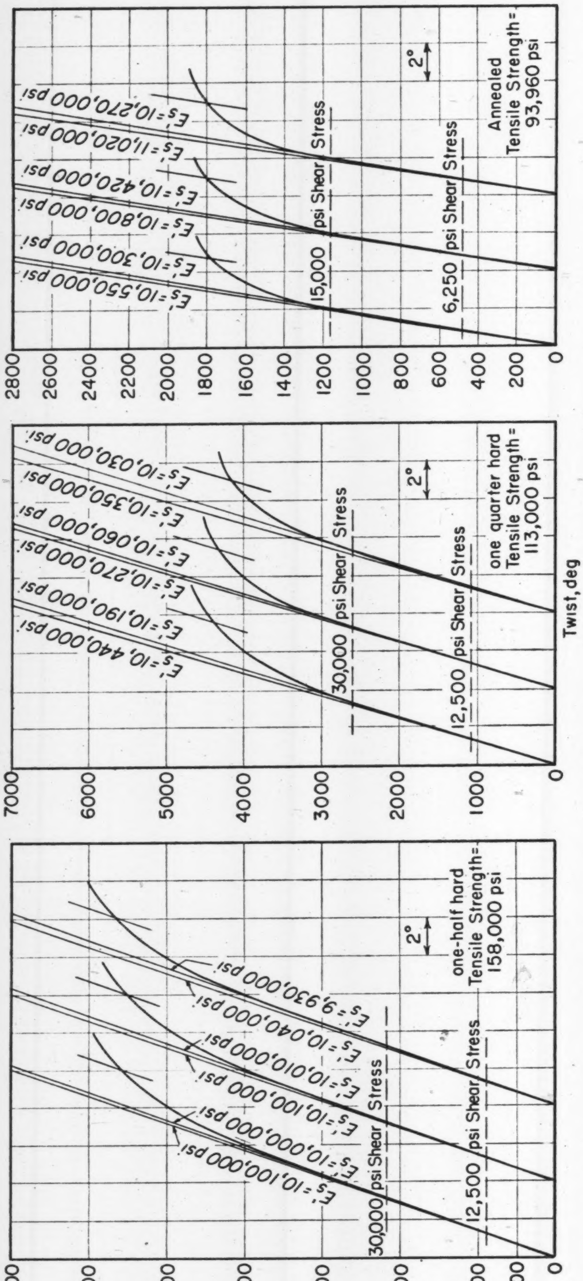


FIG. 7.—Torque-Twist Diagrams for Various Tempers of Type 347 Stainless Steel Tubing

TABLE III.—SUMMARY OF MECHANICAL PROPERTIES IN SHEAR.

Specimen Number	Tube Size, in.		Shearing Yield Strength at a Permanent Set of 2 deg per in.			Secant Shearing Modulus of Elasticity, E_s, psi				Apparent Ultimate Shearing Strength, psi	
	OD	ID	Torque, in-lb.	Shearing Yield Strength, psi	Average, psi	E_s at 12,500 psi		E_s at 30,000 psi		Individual	Average
						Individual	Average	Individual	Average		
T301-1t-1/4	0.752	0.6449	2 050	53 370	51 200	10 730 000	10 320 000	10 050 000	9 923 000	78 300	77 700
T301-2t-1/4			1 950	50 770		10 260 000		9 920 000		77 400	
T301-3t-1/4			1 900	49 470		9 960 000		9 800 000		77 500	
T301-1t-1/2	0.880	0.780	3 770	75 810	74 340	10 240 000	9 953 000	10 020 000	9 787 000	117 000	119 266
T301-2t-1/2			3 630	70 880		9 900 000		9 680 000		121 900	
T301-3t-1/2			3 910	76 340		9 720 000		9 660 000		118 900	
T302-1t-1/2	0.878	0.774	3 410	65 330	65 590	10 060 000	10 230 000	9 740 000	9 850 000	100 500	101 100
T302-2t-1/2			3 480	66 680		10 210 000		9 630 000		100 500	
T302-3t-1/2			3 380	64 760		10 410 000		10 180 000		102 300	
T304-1t-An	0.842	0.600	1 740	20 000	20 370	10 840 000[a]	11 000 000[a]	10 230 000[b]	10 490 000[b]	61 400	61 270
T304-2t-An			1 785	20 550		11 230 000[a]		10 520 000[b]		61 200	
T304-3t-An			1 785	20 550		10 920 000[a]		10 730 000[b]		61 200	
T304-1t-1/4	1.002	0.875	4 880	59 030	57 260	10 220 000	10 600 000	9 640 000	9 957 000	77 200	77 700
T304-2t-1/4			4 860	58 790		10 090 000		9 810 000		75 800	
T304-3t-1/4			4 460	53 950		11 500 000		10 420 000		80 100	
T304-1t-1/2	1.054	0.813	10 400	70 020	69 980	9 620 000	9 840 000	9 330 000	9 683 000	106 900	99 666
T304-2t-1/2			10 350	69 690		10 050 000		9 870 000		96 000	
T304-3t-1/2			10 430	70 220		9 850 000		9 850 000		96 100	
T316-1t-1/4	0.996	0.875	4 630	56 750	56 910	9 420 000	9 370 000	8 840 000	8 933 000	82 800	80 600
T316-2t-1/4			4 760	58 340		9 270 000		8 890 000		79 000	
T316-3t-1/4			4 540	55 650		9 420 000		9 070 000		80 000	
T316-1t-1/2	1.057	0.814	11 070	73 640	73 900	9 000 000	8 907 000	8 890 000	8 847 000	98 800	98 233
T316-2t-1/2			11 360	75 570		8 870 000		8 820 000		99 500	
T316-3t-1/2			10 900	72 500		8 850 000		8 830 000		96 400	
T321-1t-1/4	0.503	0.4064	628	43 900	46 740	10 110 000	10 360 000	9 420 000	9 740 000	78 600	77 666
T321-2t-1/4			713	49 850		10 580 000		9 920 000		77 200	
T321-3t-1/4			665	46 480		10 380 000		9 880 000		77 200	
T321-1t-1/2	0.502	0.4015	814	55 280	55 890	10 000 000	10 090 000	9 820 000	9 950 000	80 900	81 766
T321-2t-1/2			835	56 700		10 060 000		9 930 000		84 300	
T321-3t-1/2			820	55 680		10 220 000		10 100 000		80 100	
T347-1t-An	0.839	0.636	1 790	23 100	23 270	10 550 000[a]	10 790 000[a]	10 300 000[b]	10 330 000[b]	51 300	53 300
T347-2t-An			1 910	23 360		10 800 000[a]		10 420 000[b]		55 100	
T347-3t-An			1 910	23 360		11 020 000[a]		10 270 000[b]		53 500	
T347-1t-1/4	1.004	0.870	4 400	50 760	49 530	10 440 000	10 350 000	10 190 000	10 090 000	78 500	79 833
T347-2t-1/4			4 330	49 950		10 270 000		10 060 000		81 800	
T347-3t-1/4			4 150	47 880		10 350 000		10 030 000		79 200	
T347-1t-1/2	1.050	0.8174	11 330	78 780	78 550	10 100 000	10 080 000	10 000 000	9 980 000	90 500	93 833
T347-2t-1/2			11 180	77 740		10 100 000		10 010 000		96 900	
T347-3t-1/2			11 380	79 120		10 040 000		9 930 000		94 100	

[a] Determined at a stress of 6,250 psi.
[b] Determined at a stress of 15,000 psi.

In the derivation of this formula it is assumed that stress is proportional to strain and thus directly related to shearing moduli. The formula is therefore not strictly applicable to stainless steel because of the non-linear stress-strain relationship of this material. It is commonly used, however, as in finding the shearing stress at the yield point from a torsion test of structural steel. The relationship is actually more nearly applicable to stainless than to structural steel in determining a "yield" criteria, since the former exhibits less deviation from the straight line than the latter, which yields suddenly in the stress range beyond the proportional limit.

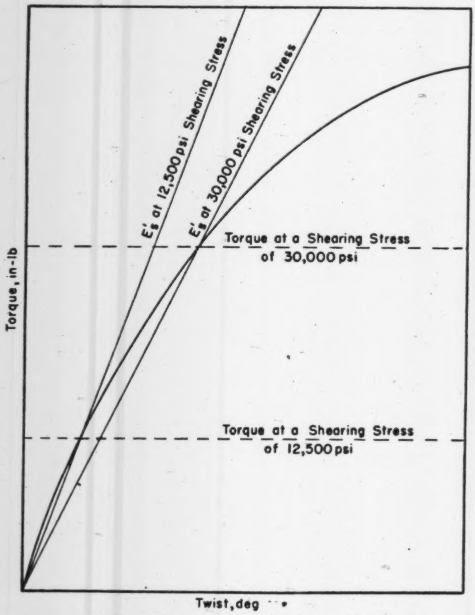


FIG. 8.—Method of Determining Secant Modulus of Elasticity at Two Stresses. Slope of line is used to calculate moduli.

The torque-twist diagrams exhibit the non-linear relationship characteristic of tensile stress-strain diagrams for stainless steel. The effect of temper on the relationships may also be seen from the figures.

Shearing Yield Strength:

Since the torque-twist relationship is non-linear, the proportional limit shearing stress is at zero. No yield point was observed, so it becomes necessary to specify a stress at a limiting amount of shearing distortion. Some authors have done this for the usual 0.2 per cent offset common for tensile yield strength determination. In the design of shafting it is common to specify the number of degrees of permanent twist which can be permitted for a particular application. The authors propose that this is a more reasonable criterion for a limiting design stress in torsion. It will be noted from Fig. 2 through 7 that a permanent angular distortion of 2 deg per in. of specimen length has been selected as the offset that determines yield strength in torsion.

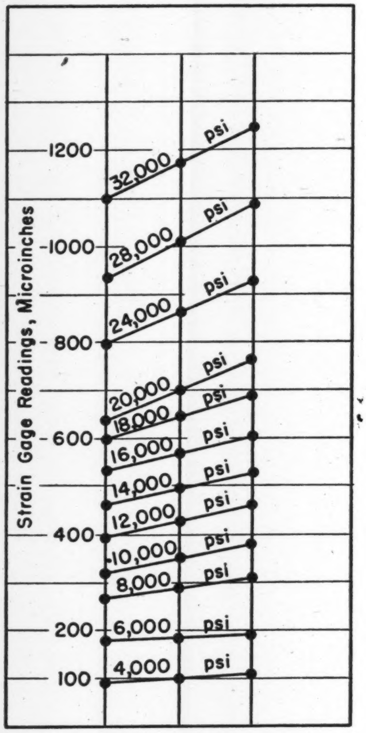


FIG. 9.—Typical Average Strain Distribution Across Face of Type 2 Poisson's Ratio Specimen For Various Stresses.

Each plotted point is the average of the readings of companion gages on opposite faces. Extreme right and left are the edges of the specimen face.

The stress is calculated from the torque obtained at the point of intersection of the curve and a line drawn parallel to the initial slope of the torque-twist diagram from a twist of 2 deg per in. Shearing yield strengths are summarized in Table III. From these data it will be noted that cold working increases the yield strength just as in tension. Thus a hard temper of any alloy has a greater shearing yield strength than a softer temper. There does not appear to be any relationship between the property and the various alloys for any given temper.

Secant Shearing Modulus of Elasticity:

The torque-twist diagrams of Figs. 2 through 7 were used to determine the secant modulus of elasticity in shear. This property was determined from the diagrams as shown in Fig. 8. A straight line was drawn from the origin to a torque equivalent to a shearing stress of either 12,500 psi or 30,000 psi as shown. The slope of this line was substituted in the following relationship to obtain the shearing modulus at this stress, identified as the secant shearing modulus.

$$E'_s = \frac{T}{\theta} \frac{584}{(d_0^4 - d_i^4)}$$

where:

E'_s = secant shearing modulus of elasticity at the specified stress, psi,

$\frac{T}{\theta}$ = slope of the secant modulus line as determined from the torque, T, in in-lb, required to produce an angular twist $\theta +$, in degrees per inch of length,

d_0 = outside diameter of tube, in., and

d_i = inside diameter of tube, in.

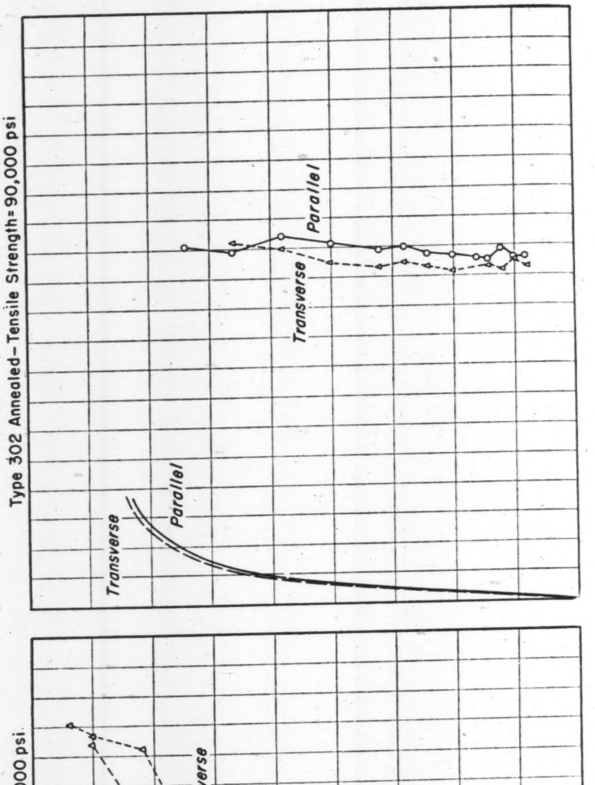
This formula is readily developed from the usual relationship in strength of materials.[3] Using the slope of the line permits more accuracy than selecting the torque and twist from a point on the diagram itself. It will be observed from Fig. 8 that the use of the secant shearing modulus to determine the angle of twist for any given torque will give values greater than actual for any stress less than that at which the modulus was obtained. For greater stresses the calculated value will be less than the actual value. A desire to develop a measure of torsional rigidity which could be obtained readily leads to the adoption of the secant rather than the tangent modulus. It is also felt that this property can be determined more accurately than the initial modulus often reported.

Table III summarizes the secant shearing moduli for shearing stresses of 12,500 psi and 30,000 psi. The lower stress permits a better comparison since the higher value may be too far into the "plastic" range. Inspection of the torque-twist diagram will show if this is the case. Because of this condition secant shearing moduli for annealed materials were calculated at stresses of 6250 psi and 15,000 psi.

The data show that shearing modulus is reduced by cold working for any given alloy. Hard tempers therefore have lower shearing moduli than softer tempers. It may also be noted that the secant shearing modulus for any temper decreases with increasing alloy number until types 321 and 347 are encountered. These alloys have higher shearing moduli than some of the soft tempers of lower series alloys. The reason for this might be found in the production method for the tubing or in the amount of free ferrite present in the tempered material.

Apparent Ultimate Shearing Strength:

When the formula previously given for shearing stress in terms of torque and specimen size is used for the maximum torque, the ultimate shearing strength can be calculated. This is a fictitious stress, however, since the formula is ap-


Type 302 Annealed-Tensile Strength = 90,000 psi
Transverse
Parallel
Parallel
Transverse
Type 301 Annealed-Tensile Strength = 102,000 psi
Transverse
Parallel
Parallel
Transverse
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
Tensile Stress, psi

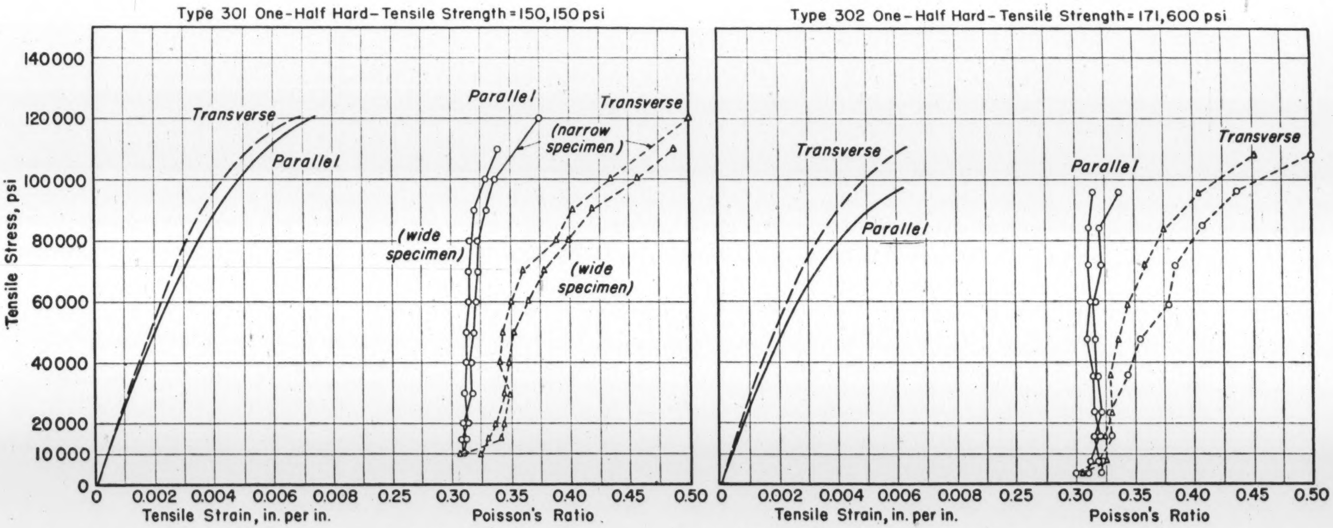


FIG. 10.—The Influence of Stress on Poisson's Ratio for Various Tempers of Types 304 and 347 Stainless Steel. Tensile stress-strain diagrams are also shown.

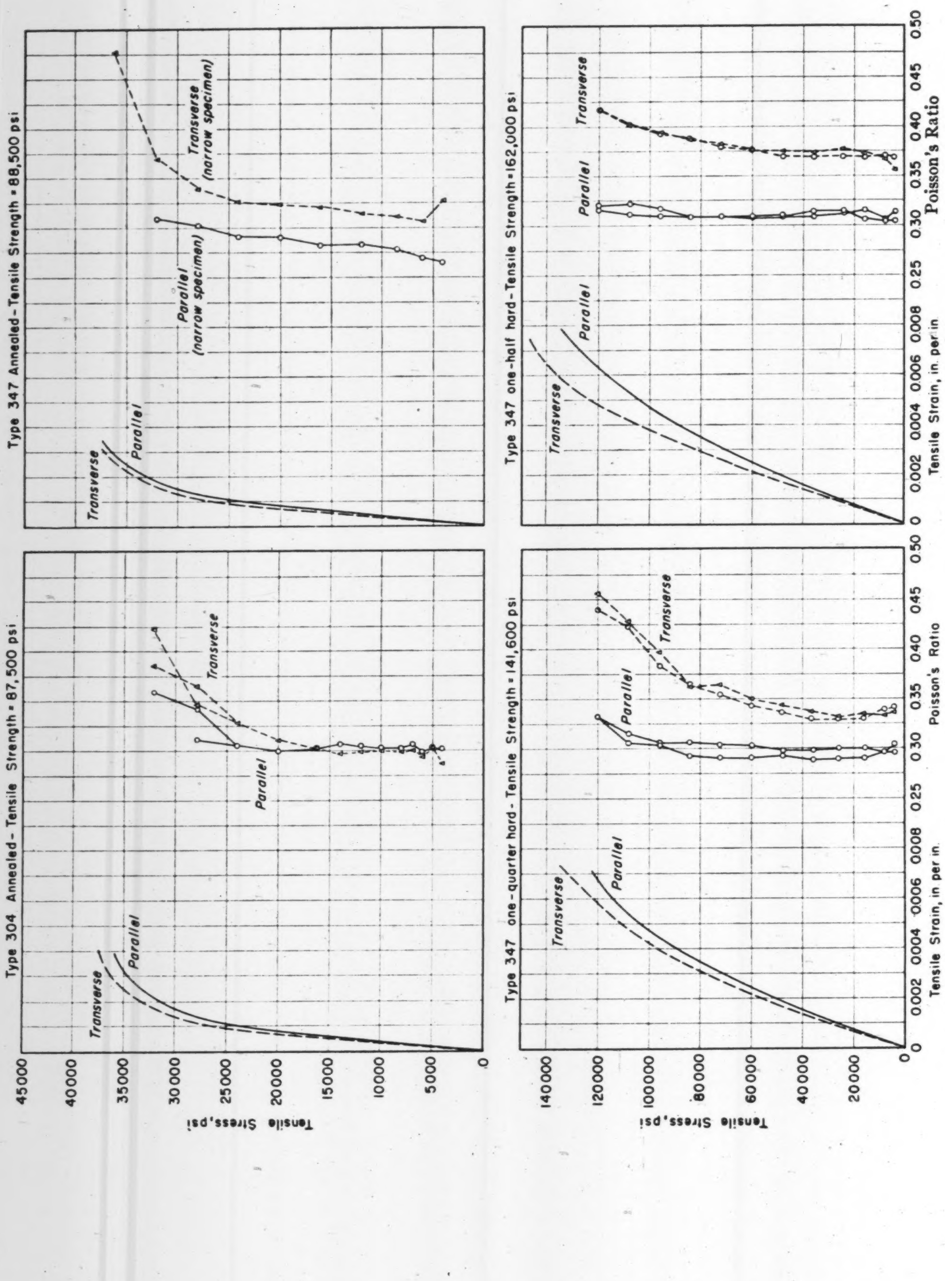


FIG. 11.—The Influence of Stress on Poisson's Ratio for Various Tempers of Types 304 and 347 Stainless Steel. Tensile stress-strain diagrams are also shown.

TABLE IV.—SUMMARY OF POISSON'S RATIO FOR VARIOUS STRESSES.

Stress, psi	Type 301-An				Type 301½				Type 302-An				Type 302½				Type 304-An				Type 347-An				Type 347¼				Type 347½			
	P1	P2	T1	T2	P1[a]	P2	T1[a]	T2	P1	P2	T1	T2	P1	P2	T1	T2	P1	P2	T1	T2	P1[a]	P2	T1[a]	T2	P1	P2	T1	T2	P1	P2	T1	T2
2 000..	0.273	0.268	0.283	0.302	...	...	...	...	0.275	0.277	0.304	0.272	...	...	...	...	0.273	0.300	0.237	0.305	...	0.447	...	...	...	...	...	...	...	...	...	...
3 000..	0.276	0.298	0.288	0.302	...	...	...	...	...	0.284	0.309	0.270	...	...	...	...	...	0.306	0.276	0.304	...	...	...	...	...	...	...	...	...	...	...	...
4 000..	0.294	0.292	0.304	0.295	...	...	...	...	0.290	0.288	0.306	0.284	0.308	0.322	0.300	0.305	0.312	0.304	0.261	0.309	0.272	0.257	0.308	0.346	...	...	...	...	...	...	...	...
5 000..	0.293	0.294	0.308	0.290	0.308	0.281	0.352	0.341	0.297	0.292	0.293	0.299	...	...	...	...	0.314	0.302	0.282	0.304	...	...	...	...	0.303	0.297	0.336	0.341	0.305	0.314	0.356	0.368
6 000..	0.300	0.292	0.308	0.290	...	...	...	...	0.297	0.290	0.294	0.301	0.304	...	...	...	0.308	0.302	0.287	0.301	0.269	0.268	0.306	0.305	...	...	...	...	...	...	...	...
7 000..	0.295	0.293	0.310	0.296	...	...	...	...	0.295	0.293	0.292	0.303	...	...	...	...	0.316	0.301	0.290	0.300	...	...	...	...	...	...	...	...	...	...	...	...
8 000..	0.301	0.300	0.310	0.296	...	...	...	...	0.296	0.296	0.289	0.305	0.312	0.320	0.319	0.325	0.312	0.302	0.292	0.304	0.281	0.272	0.308	0.313	...	...	...	...	...	...	...	...
10 000..	0.293	0.299	0.309	0.300	...	...	...	...	0.299	0.295	0.287	0.302	...	...	...	...	0.309	0.301	0.294	0.302	...	...	...	...	0.297	0.297	0.333	0.339	0.304	0.306	0.367	0.369
12 000..	0.301	0.299	0.309	0.302	0.308	0.308	0.340	0.324	0.299	0.295	0.283	0.302	0.320	...	...	...	0.308	0.302	0.300	0.298	0.278	0.287	0.305	0.323	...	...	...	...	...	...	...	...
14 000..	0.301	0.299	0.306	0.305	...	...	...	...	0.300	0.302	0.280	0.306	...	...	...	...	0.306	0.308	0.295	0.298	...	...	...	...	...	...	...	...	...	...	...	...
15 000..	...	...	...	...	0.310	0.309	0.341	0.330	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...
16 000..	0.297	0.298	0.304	0.305	...	...	...	...	0.301	0.302	0.275	0.304	0.318	0.317	0.329	0.324	0.301	0.308	0.299	0.303	0.282	0.282	0.302	0.340	0.291	0.301	0.334	0.330	0.307	0.315	0.372	0.369
20 000..	0.299	0.295	0.307	0.308	0.309	0.311	0.343	0.337	0.305	0.310	0.278	0.305	...	...	...	...	0.300	0.303	0.315	0.307	0.288	0.293	0.304	0.342	...	...	...	...	...	...	...	...
24 000..	0.301	0.294	0.313	0.308	...	...	...	...	0.314	0.312	0.283	0.314	0.316	0.321	0.328	0.330	0.306	0.310	0.331	0.326	0.290	0.292	0.307	0.345	0.291	0.301	0.331	0.329	0.314	0.312	0.376	0.369
28 000..	0.300	0.296	0.325	0.311	...	...	...	...	0.314	0.321	0.285	0.321	0.315	...	...	...	0.309	0.342	0.365	0.346	0.303	0.302	0.333	0.348	...	...	...	...	...	...	...	...
30 000..	...	...	...	...	0.315	0.311	0.348	0.345	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...
32 000..	0.311	0.298	0.330	0.332	...	...	...	...	...	0.327	...	...	...	...	...	...	...	0.358	0.386	0.423	0.313	0.306	0.369	0.372	...	...	...	...	...	...	...	...
36 000..	0.308	0.339	0.405	0.343	...	...	...	...	...	...	...	...	0.315	0.316	0.343	0.328	...	...	...	...	...	...	0.437	0.476	0.289	0.299	0.337	0.328	0.314	0.310	0.374	0.368
40 000..	0.315	0.353	0.417	0.409	0.314	0.313	0.340	0.347	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...
42 000..	...	...	0.426	...	...	...	...	...	...	...	...	...	0.315	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...
48 000..	...	...	...	...	...	...	...	...	...	...	...	...	0.310	0.316	0.354	0.336	...	...	...	...	...	...	...	...	0.294	0.299	0.344	0.337	0.310	0.309	0.375	0.368
50 000..	...	...	...	...	0.318	0.313	0.343	0.352	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...
60 000..	...	...	...	...	0.320	0.315	0.350	0.365	...	...	...	...	0.313	0.315	0.377	0.344	...	...	...	...	...	...	...	...	0.291	0.304	0.350	0.343	0.309	0.308	0.376	0.376
70 000..	...	...	...	...	0.323	0.315	0.360	0.378	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...
72 000..	...	...	...	...	...	...	...	...	...	...	...	...	0.311	0.322	0.384	0.358	...	...	...	...	...	...	...	...	0.292	0.305	0.364	0.354	0.309	0.309	0.387	0.378
80 000..	...	...	...	...	0.324	0.318	0.384	0.399	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...
84 000..	...	...	...	...	...	...	...	...	...	...	...	...	0.312	...	0.407	0.375	...	...	...	...	...	...	...	...	0.294	0.307	0.362	0.363	0.308	0.308	0.385	0.386
90 000..	...	...	...	...	0.329	0.320	0.402	0.419	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...
96 000..	...	...	...	...	...	...	...	...	...	...	...	...	0.316	0.337	0.437	0.405	...	...	...	...	...	...	...	...	0.304	0.306	0.397	0.383	0.316	0.310	0.394	0.392
100 000..	...	...	...	...	0.337	0.329	0.435	0.457	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...
108 000..	...	...	...	...	...	...	...	...	...	...	...	...	...	...	0.501	0.452	...	...	...	...	...	...	...	...	0.307	0.314	0.426	0.423	0.322	0.311	0.400	0.401
110 000..	...	...	...	...	...	0.340	...	0.487	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...
120 000..	...	...	...	...	0.375	...	0.500	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	0.334	0.335	0.456	0.440	0.318	0.317	0.416	0.416

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[a] Wide specimen.

plicable only to strain in the elastic range as has been explained previously. The values reported for this property in Table III are termed the "apparent" ultimate shearing strength. The values are analogous to the modulus of rupture for wood beams. They are reported only for comparative purposes and should not be used to calculate the ultimate strength of other sizes of tubing or for rods.

The influence of temper on apparent ultimate shearing strength is obvious from Table III. Hard tempers of any alloy have higher shearing strengths than softer tempers.

Poisson's Ratio:

Early in the investigation an effort was made to determine the best type of specimen for the Poisson's ratio studies as has been discussed. Figure 9 shows the average strain distribution across a Type 2 specimen shown in Fig. 1. Since the distribution was linear, the center gages giving the average of the outside gages, it was decided to use the Type 3 specimen for the remaining tests.

All Poisson's ratio data were carefully corrected to zero before the value itself was determined. The first step consisted of plotting the observed strain for each gage line against stress. Any points which deviated considerably from the average curve drawn through them were considered to be incorrect readings or readings taken after excessive plastic flow had occurred. Such points were corrected to place them on the curve. The curve was then checked for initial reading by extrapolating back to the ordinate intercept. If this was not the initial load, the entire curve was shifted to make the correction. This technique was applied to longitudinal and transverse readings. Values of Poisson's ratio were calculated for each stress from the relationship

Poisson's ratio = (average transverse strain corrected for Poisson effect) ÷ (average longitudinal strain)

By using this refinement, values of Poisson's ratio for duplicate specimens were found to agree within maximum of about 4 per cent. Exceptions to this were the Types 1 and 2 specimens (wide and narrow) tested in the first part of the investigation.

The relation between Poisson's ratio and stress together with average longitudinal and transverse tensile stress-strain diagrams are given in Figs. 10 and 11. The values are tabulated for closer inspection in Table IV.

There is a tendency for some alloys to exhibit a Poisson's ratio at one or two of the initial stresses that is widely different than for higher stresses. The reason for this "brooming" effect has not been determined. Reading of the gages was stopped at high stresses when the strain was beyond the range of the strain indicator.

Figures 10 and 11 show that Poisson's ratio is almost constant with increasing stress for specimens tested parallel to the direction of rolling. This is particularly interesting in view of the fact that the tensile stress-strain diagram is non-linear. The observation holds for hard or soft tempers. In the transverse direction the ratio increases with increasing stress and, with the exception of type 302 annealed, is higher than the value for the parallel direction.

Hard tempers of any of the alloys tested had higher Poisson's ratios than softer tempers in the direction parallel to rolling. In the transverse direction the same effect was observed for all alloys but type 301, where little or no difference was found.

A commonly accepted value of Poisson's ratio for stainless steel has been

TABLE V.—COMPARISON OF EXPERIMENTAL AND COMPUTED POISSON'S RATIO.

Alloy and Temper	E'_s, Average Secant Shearing Modulus of Elasticity,[a] psi	E', Average Secant Tensile Modulus of Elasticity,[a] psi	Computed Poisson's Ratio[b]	Experimental Poisson's Ratio	Percentage Variation[c]
301-1/2 hard	9 953 000	25 775 000	0.293	0.310	5.5
302-1/2 hard	10 227 000	26 600 000	0.300	0.315	4.8
304-annealed	11 000 000	28 300 000	0.287	0.305	5.9
347-annealed	10 790 000	27 830 000	0.290	0.280	3.6
347-1/4 hard	10 350 000	26 330 000	0.272	0.295	7.8
347-1/2 hard	10 080 000	25 900 000	0.285	0.309	7.8

[a] The secant shearing modulus was determined at a shearing stress of 12,500 psi for hard tempers and 6,250 psi for annealed material. The secant tensile modulus was determined at a tensile stress of 21,000 psi for hard tempers and 10,500 psi for annealed material.

[b] Based on the relationship $E'_s = \frac{E'}{2(1 + \mu)}$, from which $\mu = \frac{E'}{2E'_s} - 1$.

[c] Based on measured value being correct.

0.300, regardless of the direction of rolling. This may be satisfactory in some cases, but for type 347 one-half hard material cut transverse to the direction of rolling, for example, it would be a minimum of 25 per cent in error. Reference to Table IV will give designers the correct values. For those alloys for which Type 1 and 2 specimens were used it is suggested that the data for the narrow specimen be considered correct.

Relationship Between Secant Moduli and Poisson's Ratio:

When a material has a linear stress-strain relationship in tension, compression, and torsion, it is possible to develop a relationship between Poisson's ratio and the moduli for these conditions. This is done by an analysis of the strains or an evaluation of the energy input.[4] The resulting relationship has the form

$$E_s = \frac{E}{2(1 + \mu)}$$

where:

E_s = shearing modulus of elasticity, psi,

E = modulus of elasticity in tension and compression (considered to be the same numerically), psi,

μ = Poisson's ratio.

It was considered worth while in this investigation to check the possibility of using the secant moduli in place of the conventional moduli in the relationship above. The modified form is

$$E'_s = \frac{E'}{2(1 + \mu)}$$

where:

E'_s = secant shearing modulus of elasticity at a specified shearing stress, psi,

E' = secant tensile modulus of elasticity (taken in the direction parallel to rolling) at a tensile stress equal to 1.67 times the specified shearing stress, psi, and

μ = Poisson's ratio.

The difference in tensile and compressive moduli for orientations parallel or perpendicular to the direction of rolling was ignored. Since alloy steels generally have shearing strength properties equal to about 0.6 of their tensile counterparts, this ratio was used to select the specified stresses for secant moduli. The selected shearing stress was therefore multiplied by 1.67 to obtain its tensile equivalent. Secant shearing moduli were determined at a shearing stress of 12,500 psi for hard tempers and 6250 psi for annealed material. Equivalent tensile stresses for secant tensile moduli were 21,000 psi for hard tempers and 10,500 psi for annealed. These stress values were selected because the stress-strain relationship was comparatively straight at this point. The moduli were therefore close to the conventional initial modulus of elasticity. This can be verified by inspecting the torque-twist diagrams in Figs. 2 through 7 and the tensile stress-strain diagrams given in Figs. 10 and 11.

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[4] Joseph Marin, "Strength of Materials," pp. 224–226, Macmillan Co., First Edition, (1949).

The foregoing formula could only be checked for those alloys and tempers for which torsion and Poisson's ratio specimens were tested. Table V gives the results of these calculations. The greatest variation between computed and experimental values was 7.8 per cent. In all cases but one the computed Poisson's ratio was less than the measured value. When the formula was tried for secant shearing moduli at a shearing stress of 30,000 psi, errors as high as 42 per cent were observed, indicating that the relationship should only be used at low stresses. Obviously it would be desirable to have a complete set of secant shearing and tensile moduli with which to check the formula for a wide variety of alloys and tempers. The difficulty of obtaining some of the materials has delayed this until some future date. Until such time designers may wish to use the formula to determine Poisson's ratio for some alloys or tempers not included in this report.

TABLE VI.—TANGENT MODULUS OF ELASTICITY OF TYPE 301 AT 5000 PSI.

Temper	Tangent Modulus of Elasticity, psi			
	Longitudinal Compression	Longitudinal Tension	Transverse Tension	Transverse Compression
Full hard	26 280 000	26 400 000	29 360 000	29 320 000
3/4-hard	26 840 000	26 960 000	29 440 000	29 520 000
1/2-hard	28 440 000	28 400 000	29 360 000	29 480 000
1/4-hard	29 280 000	29 360 000	29 240 000	29 440 000
Annealed	30 200 000	30 200 000	30 200 000	30 200 000

A HYPOTHESIS FOR DETERMINING THE INFLUENCE OF COLD ROLLING ON POISSON'S RATIO FOR STAINLESS SHEET

The working of a sheet material by the cold-rolling process produces tension in the direction of rolling and compression in the transverse direction. This produces anisotropity with particular reference to the stress-strain relationship for stainless steel and thus on the modulus of elasticity.

The tangent modulus of elasticity of type 301 stainless steel at 5000 psi for various tempers is reported in Table VI.[5] From this table it will be noted that the tensile moduli for cold-rolled materials may in general be listed as follows in order of increasing magnitude:

1. Longitudinal compression (lowest modulus of elasticity)
2. Longitudinal tension
3. Transverse tension
4. Transverse compression (highest modulus of elasticity)

It will be noted that alloys of harder temper (more cold working) have lower moduli. It is a known fact that working austenitic stainless steel in a given direction will reduce the modulus in that direction. It would therefore be expected that this listing could be used as a guide to determine how Poisson's ratio would be influenced by stress and temper. Unfortunately no complete data have been published with which to show the influence of alloying elements and various tempers on the tangent modulus. The hypothesis therefore cannot be fully substantiated for all of the stainless alloys. One of the factors, for example, which may affect the results would be the amount of ferrite present in steels of various compositions as a result of cold rolling.

Figure 12 illustrates a section of a

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[5] M. Watter and R. A. Lincoln, "Strength of Stainless Steel Structural Members as Function of Design," Allegheny-Ludlum Steel Corp., pp. 21 to 37 (1950).

Poisson's ratio specimen as used in this investigation with strain gages mounted in various positions with respect to the direction of rolling of the sample. In the specimen cut parallel to the direction of rolling the transverse gage, No. 4, measures compressive strain. Since the transverse compressive modulus is the highest value in the list above, it will be suppressed by working to a harder temper. The longitudinal tensile modulus of elasticity is next to the lowest in the list above, so the longitudinal gages, No. 2, will measure a strain that is not influenced as much by cold working as the strain in the transverse direction. The transverse gage will therefore give higher strain values for hard than for soft temper materials since the modulus in that direction is reduced by cold working. This will increase Poisson's ratio. It is therefore to be expected that stainless steels of hard temper should have larger values for Poisson's ratio than for soft tempers. A study of the data in Table IV and Figs. 10 and 11 will illustrate the experimental verification of this hypothesis.

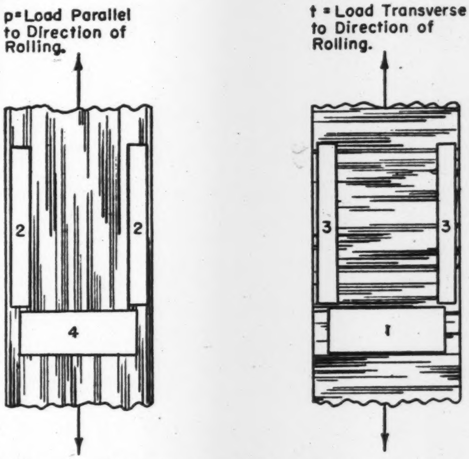
p=Load Parallel to Direction of Rolling.
2
2
4
t = Load Transverse to Direction of Rolling.
3
3
1

FIG. 12.—Influence of Direction of Prior Rolling on Poisson's Ratio.

Blocked out sections show the location of strain gages but numbers refer to order of tangent tensile modulus of elasticity in the following rating:

1. Longitudinal compression-lowest modulus
2. Longitudinal tension
3. Transverse tension
4. Transverse compression-highest modulus cold working reduces the tangent modulus so that the higher order values in this table tend inherently to move downward.

When the specimen cut in the direction transverse to the application of rolling is considered, it will be noted that the longitudinal gages, No. 3, are measuring strain governed by the modulus of elasticity in transverse tension. Since this modulus is next to the highest on the list above, it will be reduced more by cold working than will the modulus in the transverse direction for this specimen. The latter, position No. 1, represents the lowest modulus value on the list above. This means, for harder tempers, that the strain obtained from the longitudinal gages will be increased but the strain from the transverse gages will remain essentially the same. It follows, therefore, that Poisson's ratio will be smaller, or that harder tempers will have lower values for Poisson's ratio than softer tempers. However, other factors should be considered. Because the transverse gage for this condition measures strain influenced by the lowest modulus of elasticity in the list above, it is to be expected that transverse strains will be large and will change rapidly with increasing stress. This may be confirmed by referring to a typical stress-strain relationship in longitudinal compression such as given in the preceding reference. The value of Poisson's ratio may therefore actually be larger due solely to this low modulus in longitudinal compression. Only after an examination of the actual stress-strain diagrams for each temper can one predict the true influence. This analysis indicates, however, that specimens tested in the direction transverse to rolling will show a Poisson's ratio that changes with stress. An examination of Table IV will show that this is the case.

Once the fundamental relationships of the stress-strain diagram are available for any temper of any alloy, it will be possible to forecast the influence of strain hardening on Poisson's ratio following the methods set forth herein. This hypothesis appears to be a subject worth additional study.

CONCLUSIONS

The test results and the analysis presented in this report seem to warrant the following general conclusions for the stainless steels tested:

1. The torque-twist relationship is nonlinear just as in tension or compression.
2. A shearing yield strength at a permanent twist of 2 deg per in. of length provides a satisfactory limiting value for design purposes.
3. The torsional rigidity of tubing can be satisfactorily expressed by the secant shearing modulus of elasticity at low shearing stress.
4. Cold working an alloy increases the shearing yield strength and the apparent ultimate shearing strength but decreases the secant shearing yield strength. Therefore, hard tempers have higher shearing strengths and lower shearing moduli than softer tempers.
5. Poisson's ratio for investigated annealed and cold-rolled steels is almost constant with increasing stress for loads parallel to the direction of rolling, in spite of a non-linear stress-strain relationship. Poisson's ratio increases with increasing stress for loads perpendicular to the direction of rolling.
6. The commonly accepted value for Poisson's ratio of 0.300 may be satisfactory in some cases but may be 25 per cent or more in error for others.
7. Poisson's ratio for hard tempers of an alloy is somewhat higher than for soft tempers.
8. It is possible to predict the influence of cold working on the magnitude and constancy of Poisson's ratio, whether parallel or perpendicular to the direction of rolling, once the basic stress-strain diagrams are available. The hypothesis is based on the observation that the moduli are reduced by cold working.
9. The secant tensile and shearing moduli may be used to compute Poisson's ratio for low stresses. The values thus obtained compare favorably with measured values.

Acknowledgment:

This work has been carried out as a cooperative research study by the Northwestern Technological Inst. through its Faculty Research Project program and by the Development and Research Division of the International Nickel Co. The authors wish to thank the Allegheny Ludlum Steel Corp., Armco Steel Corp., Babcock & Wilcox Tube Co., Carpenter Steel Co., Superior Tube Co., and the United States Steel Co. for their kind cooperation in furnishing materials.

DISCUSSION

Mr. F. M. Howell.[1]—The authors are to be congratulated on a very fine piece of work. This is of particular interest to Committee E-1, Subcommittee 3 on Elastic Strength which has at the present time a task group working on the subject of elastic constants. Just recently they have made a survey which involved a questionnaire that was distributed to over 100 laboratories and individuals, asking about the adequacy of the information in the literature on elastic constants, and asking further about the interest in these constants.

The questionnaire indicated that the literature is woefully lacking, and this paper certainly furnishes some very interesting data on some of the stainless steels.

It would be interesting to have the authors' comments on the possible effect of residual stress in the specimens tested, upon the values of modulus and Poisson's ratio.

Mr. M. F. Sayre.[2]—I should like to ask the authors whether measurements were made of the amount of permanent set remaining after removal of the load. It seems to me that this is very important in connection with any measurements of Poisson's ratio. I have made a good many such measurements in the past, some of them on aluminum alloys, using this method of SR-4 gages in the direction of the strain and at right angles, circled around the specimen, and obtained good results.

The change observed by the authors in Poisson's ratio with increasing stress looks to me to reflect the amount of permanent set occurring. Under plastic flow, one would expect a Poisson's ratio of 0.5, and even small amounts of flow occurring would cause an approach toward this figure.

For single crystals of most metals, the tension and shear moduli vary widely in different directions, one increasing as the other decreases. As a result, in polycrystalline materials, even a slight degree of directional orientation of grains will vary the ratio between the two measured moduli and make useless the often quoted equation relating the two moduli with Poisson's ratio. Some years ago, I made careful comparative measurements of the two moduli on various spring steels in the hope of using them to determine Poisson's ratio and had this fact ground into me. I am warning my students in courses in mechanics of materials against use of this equation.

Mr. T. J. Dolan.[3]—The authors state that it was not known what method of production was used in forming the tubing. It has occurred to me that if the tubing was of the seamless-pierced variety, the production process might cause a spiral grain flow of the metal which would result in a preferred orientation of the microstructure. If all of the tests were performed with a twisting moment applied, let us say, in a clock-wise sense, a particular orientation of the maximum tensile stress with respect to the direction of grain flow is being selected.

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[2] Professor of Applied Mechanics, Union College, Schenectady, N. Y.

[3] Research Professor of Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

In the test of sheet, a certain anisotropy was found to occur with regard to Poisson's ratio and elastic properties. I am wondering whether the authors have made any tests in which the torque on the tubes was reversed, in sense, from the normal method of testing and whether this, of itself, would not change some of the values of Young's modulus of elasticity and elastic strengths observed.

I realize that this is not easy to do since most of the testing machines and instruments are adapted to one direction of rotation. It might be of some interest to see if different properties might be observed on reversing the direction of the applied torque.

MR. R. L. TEMPLIN.[4]—I should like to ask the authors what was done in their work to calibrate the troptometer and torsion machine used. Apparently, they achieved rather satisfactory sensitivity in their tests, but when determining elastic constants, some of us would like to know what the probable accuracy of the measurements is.

In their tension tests of sheet specimens, if I interpreted the data shown in one of their diagrams rightly, there is a variation of strain across the specimen of 15 per cent. We do not like that much variation in our test specimens when determining the elastic constants but try to keep such variations within about 1 per cent.

Now, from the material viewpoint, a previous discusser has raised the question about the distribution of internal strain in these specimens. In cold-worked materials, and it is not peculiar to stainless steel, we usually find rather pronounced strain gradients in the specimens, due to the methods of fabrication used.

We are, therefore, not surprised at observed differences in modulus values, when we relieve some of that internal strain in the specimens. This can be done by annealing, of course, but it is quite feasible, in the testing of specimens, to do it mechanically, and by that I mean, overstraining the specimens somewhat, then repeating the testing, and noting the modulus obtained after overstraining. One of the commoner forms of commercial relief of the strain gradients is a flattening process by stretching.

Many designing engineers insist that they are interested in the initial modulus of elasticity of a material. Usually what they want to know is the apparent modulus of some product made out of this or that material, and actually they would like to know the elastic behavior of a structure after it has withstood a certain amount of service loads. Consequently modulus values that involve, first a prestretching of some kind, or a preloading, usually are more nearly in accordance with what should be used in computing the behavior of a structure.

CHAIRMAN J. R. TOWNSEND.[5]—I would like to call attention to the fact that, under Committee E-1, Task Force Group D on Shear and Torsion Tests, under the leadership of Mr. F. S. Mapes, of the General Electric Co., was organized on March 6, and they plan to prepare a bibliography, and a questionnaire will be circulated on this subject of shear and torsion. Mr. Howell touched on this, but he did not go into detail of the actual organization. It is a very interesting subject.

MR. C. W. MUHLENBRUCH (*author*).—To answer Mr. Howell's question, regarding the effect of residual stress on the values of modulus and Poisson's ratio, and in partial answer to some of the other, there is no question but that there are residual stresses in any material produced by the common methods of tube drawing or by stretcher levelling operations.

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[5] Materials Engineer, Bell Telephone Laboratories, Inc., Murray Hill, N. J.

Whether the initial elastic properties are desirable design values, or whether they should be obtained after some forming operation takes place, is a matter too involved to discuss here.

Tubing was used for the torsion specimens, because residual stresses are less pronounced across a thin wall tube than across a solid round. Some solid rounds were tested in initial exploratory work and were found to show considerable variation, apparently due to the skin effect.

In answer to Mr. Sayre, we measured the amount of permanent set after removal of the load. It is not reported in the paper. Obviously a material having a non-linear stress-strain diagram would be expected to exhibit a permanent set upon release of load. Reloading of the specimen after removal of one cycle of load has practically no influence on Poisson's ratio for longitudinal specimens. It does, however, influence the ratio for transverse specimens. The specimens which are remaining from this investigation are currently being used to obtain the influence of cyclic loading upon Poisson's ratio, the modulus of elasticity, and the hysteresis effects. I hope to present that information at an early date.

I find that round specimens, using four pairs of longitudinal and transverse gages make very fine specimens for obtaining Poisson's ratio in both tension and compression. However, the influence of the tube production method is very important there, because of the method of producing the tubing. The paper points out very carefully that the relationship between the tensile and torsional moduli is applicable only for those stress ranges for which it is tried in the paper and only for low stresses, at that. At the second limiting stress, the error is 42 per cent, not 7.8 per cent, as given in the paper.

In answer to Mr. Dolan, we have not tried to reverse the direction of torque. This is an interesting proposal that will be tried sometime.

In answer to Mr. Templin, troptometer used calibrates to about $\frac{1}{2}$ to 1 per cent.

The variation in strain across the specimens was undoubtedly due to the fact that they had residual stresses, and also to the possibility that it came from either coil or strip. When the 26 in. long specimen was cut from coil, it has some bow, perhaps as much as 1 in. in its length.

In such cases the specimen was held on a flat surface with weights. The strain gages were glued on one side and allowed 24 hr drying time. The specimen was turned over, flattened again, and the strain gages glued on the other side.

The 15 per cent variation in strain estimated across the specimen shown is due partly to that particular condition and partly to residual stress. It is not likely that any other instrumentation would have changed this situation.

In regard to use of the overstraining and technique, this cannot be done with a material having a non-linear stress-strain relationship, because the virgin stress-strain diagram is very quickly disturbed with one cycle of load. Stainless steel, therefore, has the unique property, that once it is loaded, it is no longer the original material.

I would like to ask Mr. Mayne whether he will explain the manner of tube production.

MR. C. R. MAYNE (*author*).—Mr. Dolan questioned the effect of the grain orientation which was in the tubing due to the piercing operation. The tubing which we used, with the exception of Type 302 which was welded, was seamless tubing and had been subjected to the piercing operation. We obtained the seamless tubing from three sources, namely, B. & W Tube Co., National Tube Co., and Superior Tube Co. The first two pierce their own tubes and Superior produces their product from tubes pierced by others. The piercing operations conducted by the first two are carried out with the rotation of the billets in opposite directions. Thus our torsion tests were made partially with the torque applied in the same direction as the twist during piercing and partially with the torque applied opposite to the twist of piercing. In fact, the annealed Types 347 and 304 were obtained from one of these two sources and the hard drawn tempers from another. Although we did not note the direction of orientation of grains due to the piercing operation, it would seem that the results are not affected by any orientation caused by the hot working operation.

In regard to the method of production of the tubing used in these experiments there were three. First, some of the tubing was produced on tube reducing machines. The second method of production was by plug drawing and the third method was by bar drawing. In the first method the tubing becomes quite warm and probably the amount of ferrite is limited by the heat produced during cold working. We do not know whether or not the amount of ferrite will influence the results. The third method of production involves drawing the tubing down onto a solid mandrel and then expanding the tubing a few thousandths to withdraw the bar mandrel. We did not investigate the effect of production method on the results of torque twist tests but we do feel that the effect would be rather small.

CREEP-RUPTURE OF SEVERAL SHEET STEELS*

By G. V. Smith,[1] E. J. Dulis[1] and E. G. Houston[1]

Synopsis

Grips for testing sheet or strip in creep-rupture are described and experimental results reported for the following materials and test temperatures: (1) annealed 18/Cr, 8/ Ni, (Mo) at 1100, 1300, and 1500 F; (2) annealed and half-hard 17/Cr, 7/Ni at 600 and 1100 F; and (3) cold-rolled 4608 (Ni, Mo) at 600 F.

Although many applications at elevated temperature involve metals in the form of sheet or strip, the great majority of creep and creep-rupture data have been obtained on bar or plate stock. It is well known that the properties of sheet may differ significantly from bar or plate at ordinary temperature, owing to differences in processing, and since the creep and rupture properties are even more structure-sensitive than the room temperature properties, it may be expected that differences in processing may yield even greater differences in creep properties. In addition, a few experimental data obtained by Thielemann and Parker (**1**)[2] have indicated that the atmosphere in which the creep-rupture test is made may affect the strength. Inherent in such an effect is the influence of size and shape of specimen, that is, the ratio of surface area to volume.

Materials and Apparatus

The chemical composition of the steels studied is given in Table I. The 18 Cr, 8 Ni, (Mo) (AISI Type 316) steel had been hot rolled to 0.050-in. gage, annealed and pickled; the annealing treatment was 6½ min at 1975 to 2000 F and air-cooled. The 17 Cr, 7 Ni (AISI Type 301) steel was supplied in 0.050-in. sheet in both the annealed and half-hard conditions; the annealed material had been heated 6½ min at 1950 F, air-cooled and descaled, while the half-hard material received a final cold rolling of about 25 per cent reduction in thickness after such annealing. All materials were tested in the longitudinal direction.

In designing grips for creep-rupture specimens from sheet stock, it was considered desirable that the gripping should be effected within the furnace, in order to conserve test materials and to permit testing of samples available only in limited lengths. As shown in Fig. 1, the specimen is gripped between wedges and is also supported by pins through the wedges and specimen. The wedges do not have serrated surfaces, and the pins are required to minimize slipping, especially during application of the load. Even so, some slipping, as well as deformation at the pins, occurred, so that the extensions measured autographically during test by the fall of the lever (**5**) are only approximate.[3] All parts of the grips are made of

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*Presented at the Fifty-fourth Annual Meeting of the Society, June 18–22, 1951.

[1] Research Laboratory, United States Steel Co., Kearny, N. J.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 868

[3] For this reason, we have, since this work was completed, adopted a wire-tube extensometer similar to that described by Fellows and associates (6), viewed through a window in the test furnance.

25 Cr, 20 Ni, 4 W stainless steel, an arbitrary selection which has given satisfactory service for pull-rod and similar applications since the inception of creep testing in this laboratory. The test stands are identical with those described earlier (5) except that it was necessary to counterbalance the weight of the loading lever by a front extension in order to permit sufficiently low test loads for sheet materials which have relatively small cross-sectional area.

TABLE I.—CHEMICAL COMPOSITION OF STEELS.

Steel Type	Heat	C	Mn	P	S	Si	Ni	Cr	Mo
316..................	No. 7X1109	0.046	1.53	0.022	0.010	0.32	12.34	16.94	2.20
309..................	No. X13196	0.09	0.61	0.023	0.010	0.27	7.49	17.35	...
4608..................	No. 7X4609	0.09	0.35	0.010	0.010	0.13	1.57	...	0.21

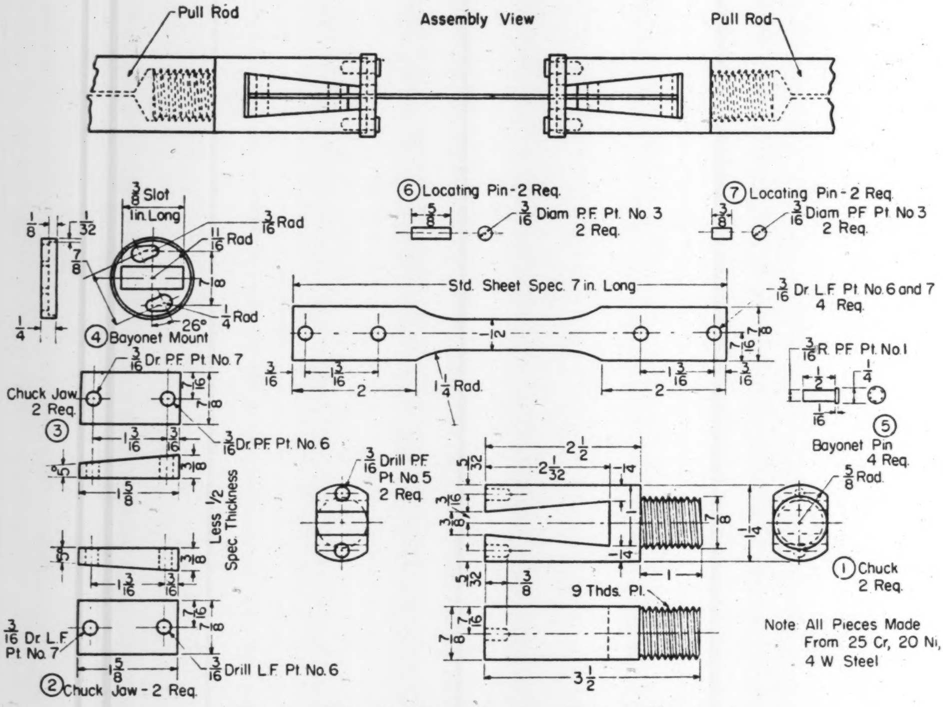

Pull Rod
Assembly View
Pull Rod
3/8 Slot 1 in. Long
3/16 Rad
11/16 Rad
1/4 Rad
26°
④ Bayonet Mount
⑥ Locating Pin - 2 Req.
3/16 Diam P.F. Pt. No 3 2 Req.
⑦ Locating Pin - 2 Req.
3/16 Diam PF Pt No 3 2 Req.
Std. Sheet Spec. 7 in. Long
3/16 Dr. L.F. Pt. No. 6 and 7 4 Req.
1 1/4 Rad.
3/16 Dr. P.F. Pt. No. 7
Chuck Jaw 2 Req.
③
3/16 Dr. P.F. Pt. No. 6
3/16 R. P.F. Pt. No. 1
⑤ Bayonet Pin 4 Req.
5/8 Rad.
3/16 Drill P.F. Pt. No. 5 2 Req.
Less 1/2 Spec. Thickness
9 Thds. Pl.
① Chuck 2 Req.
3/16 Dr. L.F. Pt. No. 7
3/16 Drill L.F. Pt. No. 6
② Chuck Jaw - 2 Req.
Note: All Pieces Made From 25 Cr, 20 Ni, 4 W Steel

FIG. 1.—Grips for Creep Rupture Tests of Sheet.

RESULTS AND DISCUSSION

Mechanical Tests:

The results of short-time tension tests of four materials at various temperatures are summarized in Table II. Results of the tests on 18 Cr, 8 Ni (Mo) sheet at 1100, 1300, and 1500 F are plotted in Fig. 2. The relations observed are similar in character to those observed for bar stock (2). The stress to cause a creep rate of 0.01 per cent per hr and the stress to cause rupture in 1000 hr are given in

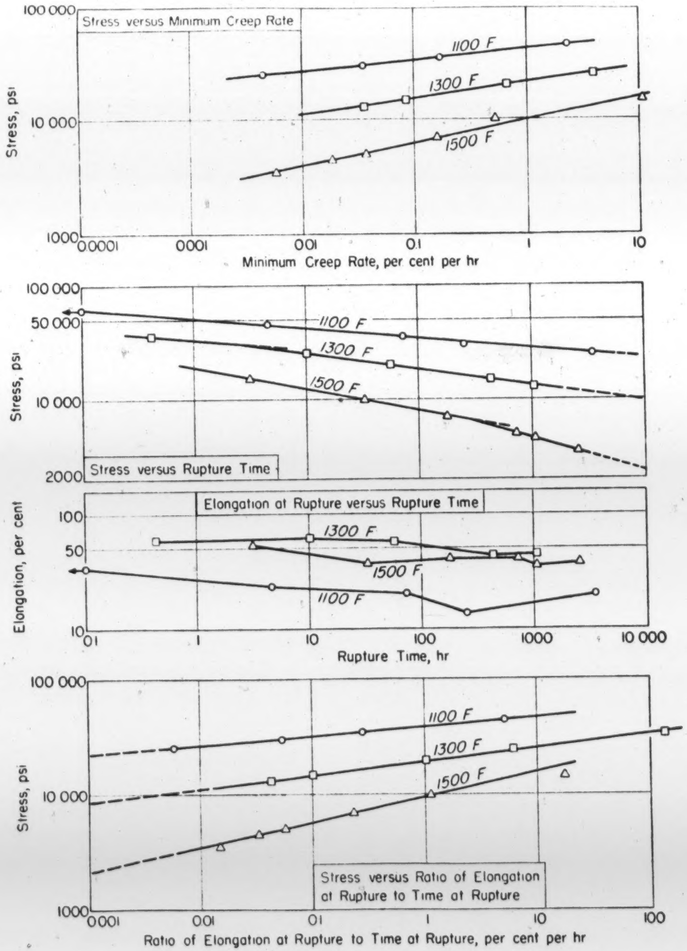


FIG. 2.—Properties of 18 Cr, 8 Ni (Mo) Sheet Steel in Creep-Rupture Tests at 1100 F, 1300 F, and 1500 F.

TABLE II.—SHORT-TIME TENSILE PROPERTIES OF SHEET STEELS.[a]

All tests parallel to rolling direction.

Test Temperature, deg Fahr	75 F	600 F	1100 F	1300 F	1500 F
18 Cr, 8 Ni (Mo) (316) ANNEALED					
Yield strength, 0.2 per cent offset, psi	33 300	21 000	16 000	14 500	13 000
Tensile strength, psi	81 000	64 000	53 000	39 000	24 000
Elongation in 2 in., per cent	57.0	36.0	40.0	50.0	60.0
17 Cr, 7 Ni (301) ANNEALED					
Yield strength, 0.2 per cent offset, psi	40 000	23 500	18 000	...	...
Tensile strength, psi	118 000	66 500	53 000	...	...
Elongation in 2 in., per cent	68.5	36.0	36.0	...	...
17 Cr, 7 Ni (301) HALF-HARD					
Yield strength, 0.2 per cent offset, psi	128 000	110 000	64 000	...	...
Tensile strength, psi	166 200	131 000	82 000	...	...
Elongation in 2 in., per cent	28.0	5.0	11.0	...	...
4608 (COLD ROLLED)					
Yield strength, 0.2 per cent offset, psi	97 100	87 900	...	...	...
Tensile strength, psi	104 600	99 300	...	...	...
Elongation in 2 in., per cent	8.5	8.3	...	...	...

[a] Except for 4608 steel and room temperature results, which represent actual tests, these data were obtained by interpolation of test data obtained for these same steels at other temperatures.

TABLE III.—CREEP-RUPTURE PROPERTIES OF 18 Cr, 8 Ni (Mo) SHEET STEEL.

Property	1100 F	1300 F	1500 F
Stress for minimum creep rate of 0.01 per cent per hr, psi	26 000	11 000	4 000
Stress for rupture in 1000 hr, psi	28 000	13 000	4 500

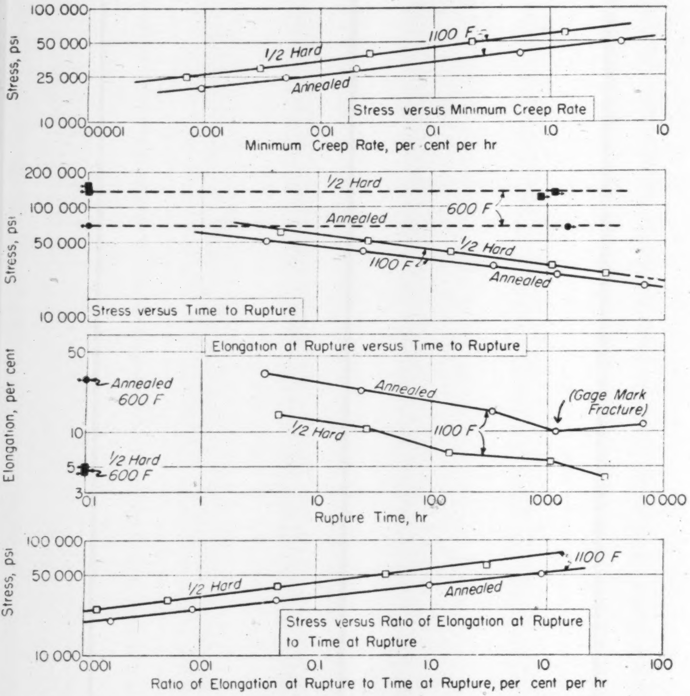


FIG. 3.—Properties of Annealed and Half Hard 17 Cr, 7 Ni Steel in Creep-Rupture Tests at 600 F and 1100 F.

Table III. The plot of elongation at rupture *versus* rupture time is rather irregular, for the sheet as observed for bar material (2). It is of interest to note that, for the same rupture time less elongation is observed at 1100 F than at 1300 or 1500 F, which was also true of the bar material (2). The final plot of stress against ratio of elongation at rupture to time at rupture permits the estimation of ductility for times beyond those tested, as described in a previous publication (2).

The test results for the annealed and half-hard 17 Cr, 7 Ni steel at 600 and 1100 F are shown in Fig. 3. Test points are few at the 600 F level; specimens either broke immediately on application of load or, under only 5000 psi less stress, had not yet broken when discontinued at 1000 hr or longer. Moreover, these breaking stresses are of the same order as the short-time tensile strength. Thus it would appear that for most applications at this temperature creep need not be considered (3).

At 1100 F, the half-hard 17 Cr, 7 Ni material is significantly stronger than the annealed in respect to both the stress to cause a specific minimum creep rate and the stress to cause fracture in a specific time, within the range investigated; actual values for these characteristics are given in Table IV. The curves of stress *versus* rupture time appear to approach one another at long time. Cold

TABLE IV.—CREEP-RUPTURE PROPERTIES OF ANNEALED AND HALF-HARD 17 Cr, 7 Ni SHEET STEEL.

Property	600 F		1100 F	
	Annealed	Half-Hard	Annealed	Half-Hard
Stress for minimum creep rate of 0.01 per cent per hr, psi	65 000	130 000	26 000	35 000
Stress for rupture in 1000 hr, psi	65 000	130 000	25 000	30 000

TABLE V.—RESULTS OF CREEP-RUPTURE TESTS OF COLD-ROLLED 4608 STEEL AT 600 F.

Stress, psi	Time to Rupture, hr	Minimum Creep Rate, per cent per hr	Elongation at Fracture, per cent
95 000	2.1	1.05	5.5
80 000	Discontinued at 4440 hr		
90 000	Discontinued at 2140 hr		

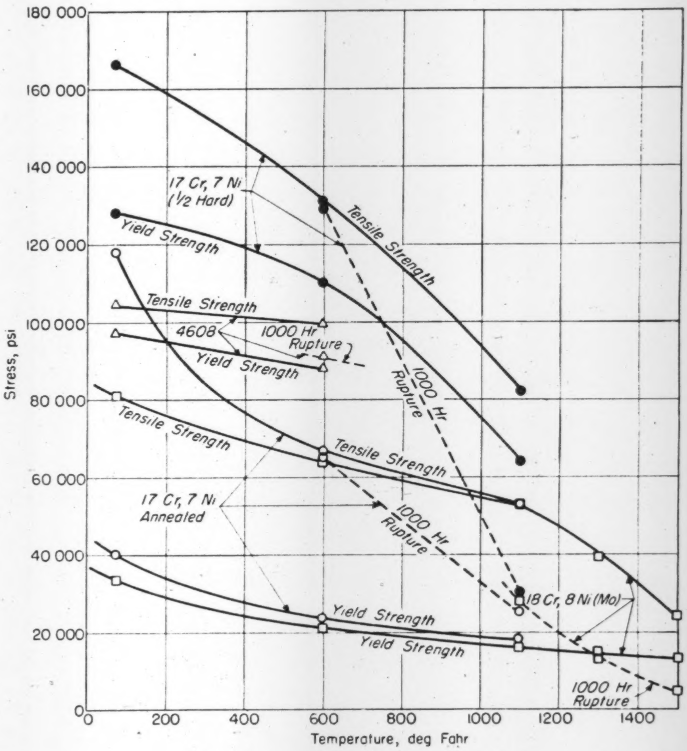
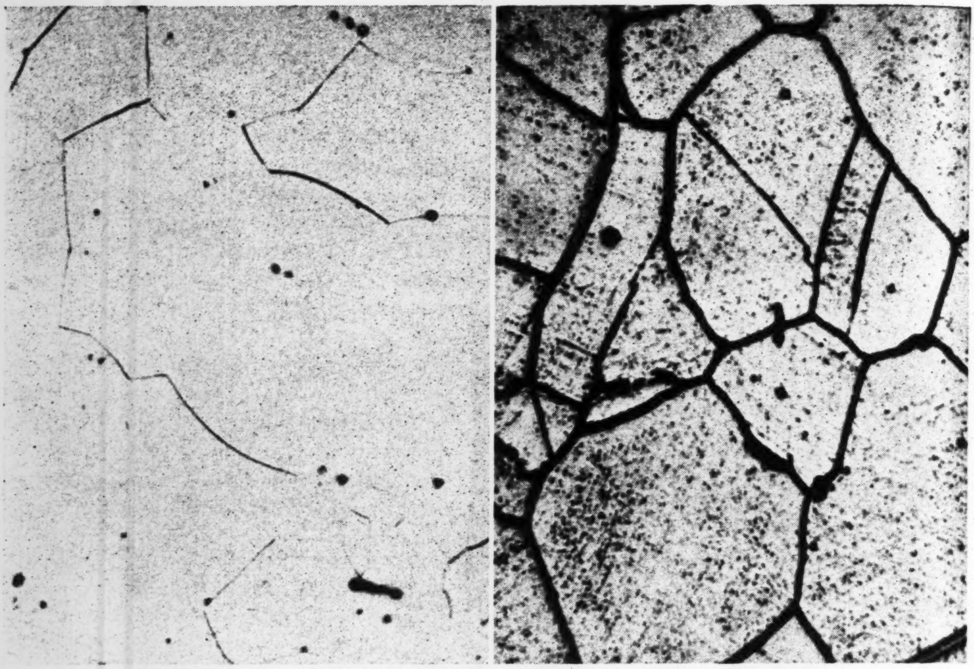


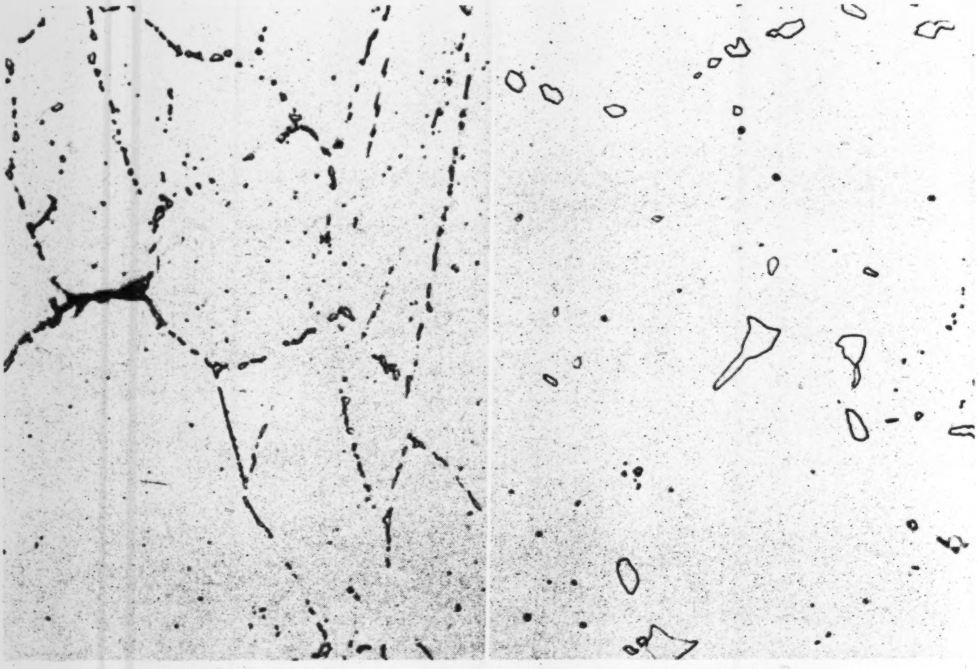
FIG. 4.—Variation of Strength With Temperature for Sheet Steels Studied in This Investigation.

working is an effective means of strengthening this material for service at temperatures at least as high as 1100 F and for periods within the range investigated. At sufficiently high temperature and possibly at sufficiently long time, this superiority should vanish owing to recovery or recrystallization of the cold-worked material before or during test.

The elongations of both the annealed and the half-hard 17 Cr, 7 Ni materials decreased with increasing time for rupture at 1100 F (Fig. 3) and in the case of the half-hard, which was less ductile, had decreased to 4 per cent on rupture in 3000 hr. The elongations of all 17 Cr, 7 Ni specimens broken at 600 F corresponded with those observed in the short-time hot-tension test at the same temperature (Table II). Attention is directed to the low elongation of the half-hard material at this temperature. The final

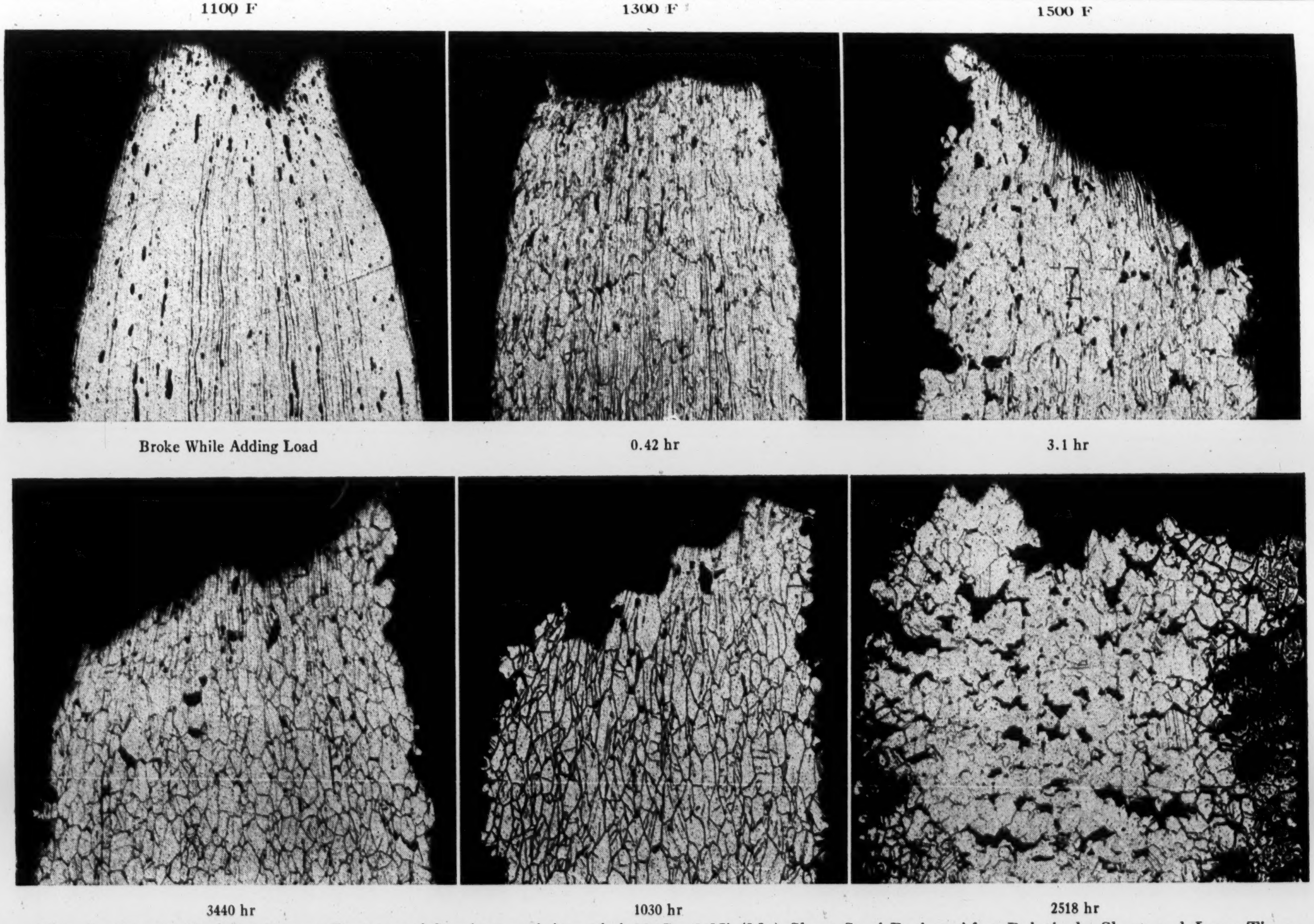


(*a*) Before Test—Annealed (*b*) 1100 F—3440 hr



(*c*) 1300 F—1030 hr (*d*) 1500 F—2520 hr

FIG. 5.—Microstructure of Annealed 18 Cr, 8 Ni (Mo) Sheet Steel Before and After Creep-Rupture Test for Times and at Temperatures Indicated. Etched in Picric-HCl Acids in Alcohol (× 1000).

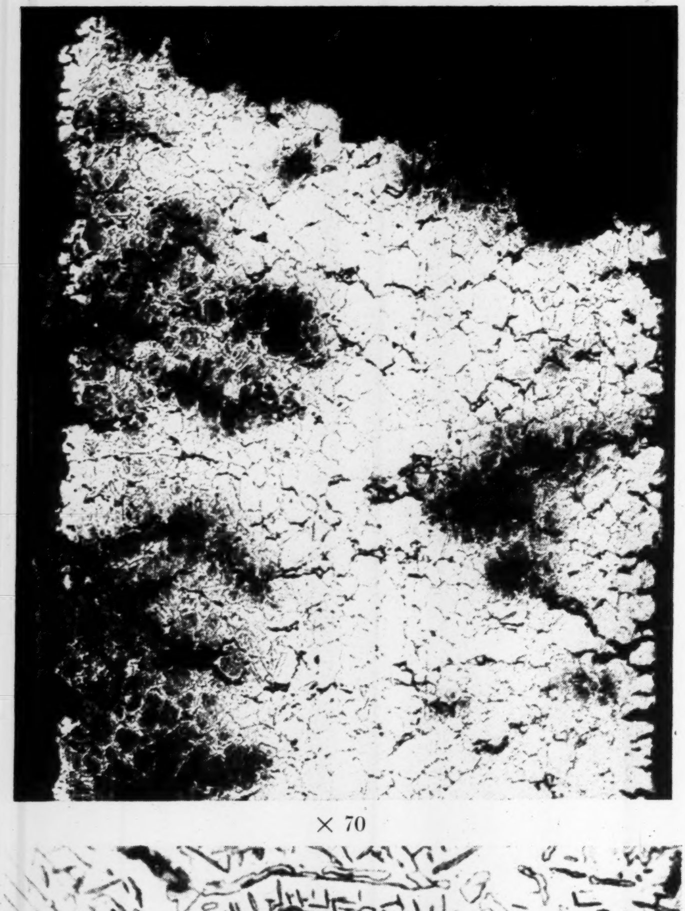


1100 F | 1300 F | 1500 F

Broke While Adding Load | 0.42 hr | 3.1 hr

3440 hr | 1030 hr | 2518 hr

FIG. 6.—Longitudinal Sections at Fracture of Specimens of Annealed 18 Cr, 8 Ni (Mo) Sheet Steel Broken After Relatively Short and Long Times. Rupture Times and Test Temperatures are Indicated. Etched in Picric-HCl Acids in Alcohol (× 70).



× 70

× 1000

Fig. 7.—Edge of 18 Cr, 8 Ni (Mo) Sheet Specimen Tested at 1500 F for 2518 hr Showing Extent and Structure of Widmanstätten Constituent Assumed to be Result of Nitrogen Penetration into the Steel. Etched in Picric-HCl Acids in Alcohol.

part of Fig. 3 shows a linear relation on log-log coordinates between stress and ratio of elongation at rupture to time at rupture, which is useful (2) for estimating elongation at fracture times beyond those tested.

The results of the tests on cold-rolled 4608 steel at 600 F are presented in Table V. The behavior of this material was similar to that of the 17 Cr, 7 Ni steel at this temperature, namely, a stress corresponding to the short-time tensile strength at this temperature was required to break the specimen, whereas a stress approximately only 10,000 psi less did not cause rupture in 2000 hr at which time the test was discontinued. Thus, this steel could be used in most applications at this temperature without consideration of creep.

The results of the tests reported herein are summarized and may be compared in the plot of Fig. 4.

Microstructural Studies:

Fractured specimens of the 18 Cr, 8 Ni (Mo) steel tested at 1100, 1300, and 1500 F and of the annealed and half-hard 17 Cr, 7 Ni steel tested at 1100 F were sectioned longitudinally and examined for structural changes and fracture characteristics. No examination was made of the 600 F test specimens which are assumed to correspond closely to room temperature behavior in these respects.

The microstructural changes observed in the 18 Cr, 8 Ni (Mo) steel, illustrated in Fig. 5, are similar to those observed in bar stock and reported previously (2), except that at 1100 F there is a suggestion of precipitation on slip planes in the sheet, possibly owing to the greater deformation. At 1100 F, a fine precipitate, judged to be largely carbide but possibly containing some sigma or chi phase[4] is observed, whereas at 1300 and 1500 F, with increasingly agglomerated particles, sigma or chi and carbide are observed. These phases are not differentiated in the photomicrographs since no single etchant which will do this has been discovered (4).

The type of fracture observed in the 18 Cr, 8 Ni (Mo) sheet specimens is illustrated in Fig. 6 for the shortest and longest rupture times experienced in the tests at each of the temperatures examined. The short-time fractures are characterized by severely elongated grains and a transgranular fracture path. With increasing time for failure and increasing temperature, the grains become less distorted at the fracture, even though considerable over-all extension occurred, and the fracture path is increasingly along the grain boundaries, that is, intergranular. The latter is also apparent by the general intergranular cracking at some distance from the fracture, as especially apparent in the long time fracture at 1500 F.

Near the edge of the specimen of 18 Cr, 8 Ni (Mo) sheet which fractured in the longest time at 1500 F, and associated with the intergranular edge cracks, a Widmanstätten structure is observed (Fig. 7). On the basis of observations and chemical analyses made on bar stock and previously reported (2), this structure is attributed to the absorption of atmospheric nitrogen during test. The structure is especially plentiful in the sheet as contrasted with the bar stock, as a consequence, presumably, of the greater ratio of surface to volume.

Examination of the annealed and half-hard 17 Cr, 7 Ni steel after creep-rupture test at 1100 F, and comparison with the untested material showed the precipitation of carbide (associated with the slip markings in the half-hard material), and a change in amount of ferrite but no evidence of recrystallization. The microstructures near the fracture of the speci-

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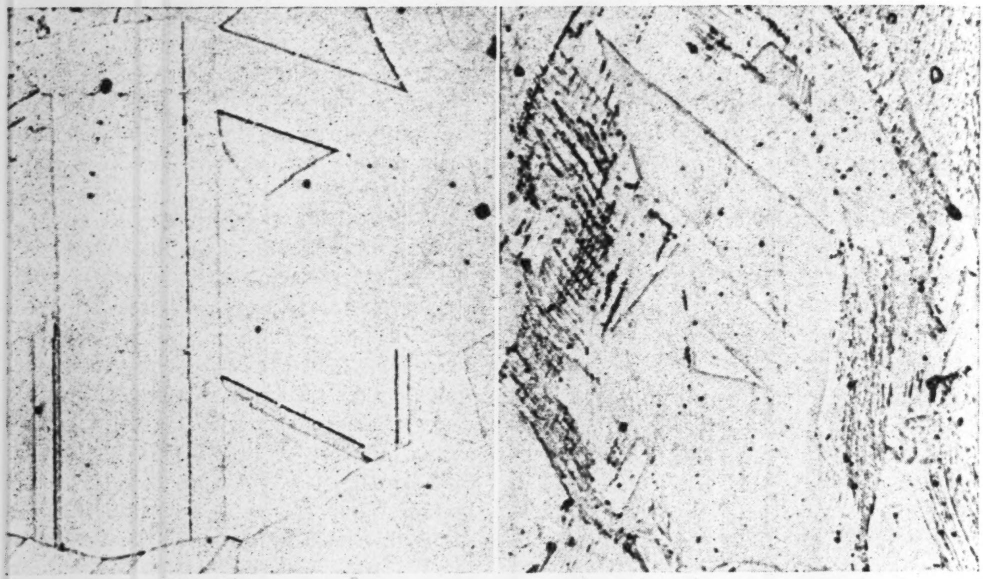
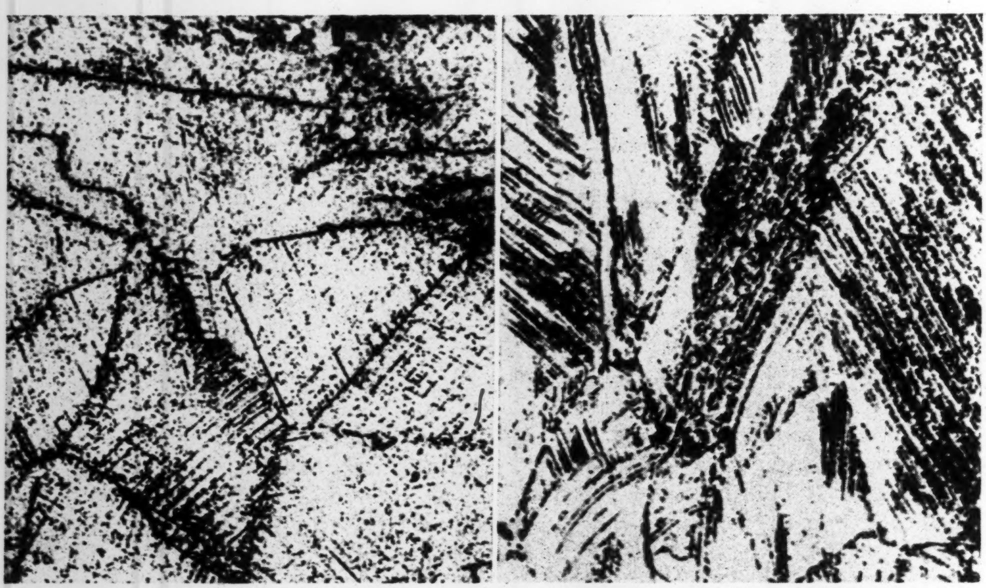
[4] Sigma and chi phase recently reported by Andrews (7) are known to form in type 316 steel, the occurrence and quantity of each being dependent on time and temperature.



(*a*) **Before Test—Annealed** (*b*) **Before Test—Half Hard**



(*c*) **Initially Annealed Tested 1100 F—6622 hr** (*d*) **Initially Half Hard Tested 1100 F—3084 hr**

FIG. 8.—Microstructure of 17 Cr, 7 Ni Sheet Steel Before and After Creep-Rupture Tests at 1100 F for the Times Indicated. Etched Electrolytically in 10 per cent Chromic Acid (× 1000).

mens which required the longest times for rupture are shown in Fig. 8 in comparison with the original material; specimens were electrolytically polished in $H_2SO_4 \cdot H_3PO_4 \cdot CrO_3 \cdot H_2O$ solution and electrolytically etched in 10 per cent chromic acid. No evidence of recrystallization was detected. The change in type of fracture from transgranular to intergranular with increase in the time for rupture was clearly apparent in the annealed material, but in the half-hard material the change, while it appeared to be tending to occur, was difficult to detect owing to the elongated character of the grains in the initial material.

TABLE VI.—X-RAY DETERMINATION OF FERRITE IN ANNEALED AND HALF-HARD 17 Cr, 7 Ni SHEET STEEL BEFORE AND AFTER CREEP RUPTURE TEST AT 1100 F.

Initial Condition	Rupture Time, hr	Temperature, deg Fahr	Ferrite, per cent[a]
Half-Hard	Before creep test	...	36
	4.6	1100	20
	26.5	1100	17
	139.0	1100	28
	139.0	1100	28
	1034.0	1100	18
	3084.0	1100	25
Annealed	Before creep test	...	0
	3.5	1100	8
	24.0	1100	16
	328.0	1100	31
	1143.0	1100	40
	6622.0	1100	59

[a] Per cent Ferrite = $\dfrac{I_F}{I_F + I_A}$

where I_F = Intensity of diffraction from (110) planes of ferrite,

and I_A = Intensity of diffraction from (111) planes of austenite.

The effect of exposure and creep deformation at 1100 F on the amounts of ferrite and austenite, which are difficult to differentiate by etching, was determined by X-ray diffraction. The measurements were made at the surfaces of the 17 Cr, 7 Ni samples about ½ in. from the fractures using a recording spectrometer with Cr K-alpha radiation. The results are tabulated in Table VI. The amount of ferrite in the annealed material increases continuously from 0 to about 60 per cent with increase of the rupture time to 6600 hr. The amount of ferrite in the half-hard material was greatest before test and decreased during creep-rupture test; the change with time for rupture appears erratic and may not be significant. Whether the ferrite in the initially annealed material formed during test or during subsequent cooling is not known. Experiments on other austenitic stainless steels indicate that, in some cases, ferrite develops during cooling after exposure at elevated temperature.

SUMMARY

Creep-rupture tests have been made on the following sheet steels: annealed 18 Cr, 8 Ni (Mo) at 1100, 1300 and 1500 F; annealed and half-hard 17 Cr, 7 Ni at 600 and 1100 F; and cold-rolled 4608 (Ni, Mo) at 600 F.

Test results in the longitudinal direction of annealed and half-hard 17 Cr, 7 Ni steel at 600 and 1100 F show appreciable superiority of the cold-worked metal at 600 F and for relatively short rupture times at 1100 F. With increase of rupture time at this latter temperature, the superiority of the cold-worked metal is diminished, though still evident at 5000 hr.

The test results on 17 Cr, 7 Ni and 4608 at 600 F suggest that creep data are probably not needed for design in either case.

Acknowledgment:

The short-time tension test data reported herein were obtained by W. B. Seens and W. L. Jensen. The sheet grips for the creep-rupture tests were made under the supervision of Q. Henderson. The test steels were provided by R. F. Miller and P. R. Wray of United States Steel Co.

REFERENCES

(1) R. H. Thielemann and E. R. Parker, "Fracture of Steels at Elevated Temperatures After Prolonged Loading," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 135, pp. 559–582 (1939).

(2) G. V. Smith, E. J. Dulis and E. G. Houston, "Creep and Rupture of Several Chromium-Nickel Austenitic Stainless Steels, *Transactions*, Am. Soc. Metals, Vol. 42 (1950), pp. 935–980.

(3) G. V. Smith, W. B. Seens and E. J. Dulis, "Hardened Alloy Steel for Service up to 700 F," *Proceedings*, Am. Soc. Testing Mats., Vol. 50, pp. 882–892 (1950).

(4) E. J. Dulis and G. V. Smith, "Identification and Mode of Formation and Re-Solution of Sigma Phase in Austenitic Fe-Cr-Ni Steels. Symposium on the Nature, Occurrence, and Effects of Sigma Phase, Am. Soc. Testing Mats., p. 3 (Symposium issued as separate publication, *STP No. 110*).

(5) G. V. Smith, W. G. Benz and R. F. Miller, "Creep and Creep-Rupture Testing," *Proceedings*, Am. Soc. Testing Mats., Vol. 47, pp. 615–638 (1947).

(6) J. A. Fellows, E. Cook and H. S. Avery, "Precision in Creep Testing," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 150, pp. 358–372 (1942).

(7) K. W. Andrews, "A New Intermetallic Phase in Alloy Steel," *Nature*, December 10, 1949, p. 1015.

DISCUSSION

MR. HENRY PAIGE.[1]—I wonder whether the Widmanstätten pattern of the chromium precipitate was found in any sheet material exposed to high temperature and not subjected to any stresses. Does the precipitation require temperature and stress or will temperature alone suffice?

MR. E. J. DULIS (*author*).—We have not investigated the effect of stress on the Widmanstätten precipitation of chromium nitride.

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[1] Metallurgist, Aeronautical Materials Laboratory, Naval Air Material Center, Philadelphia, Pa.

THE FABRICATION OF CHROMIUM-MOLYBDENUM STEEL PIPING FOR 1000 TO 1050 F CENTRAL STATION SERVICE*

By R. W. Emerson[1]

Synopsis

This paper presents a brief background relative to the sequence of events which led to the use of the steam piping materials currently being specified.

Certain metallurgical characteristics of the low-carbon chromium-molybdenum alloys are presented which indicate the possibilities of heat treating such material to a wide range of mechanical properties, depending upon the type of heat treatment specified.

A discussion of the factors which are pertinent in the heat treatment of alloy steel piping to obtain good high-temperature properties is given and several specific questions are raised relative to heat treatment for obtaining best high-temperature properties at 1000 and 1050 F.

Data on the structural stability of the 2¼ per cent chromium, 1 per cent molybdenum alloy is presented together with impact test data both before and after 2000 hr at 1050 and 1150 F.

A discussion and data on the welding of 2¼ per cent chromium, 1 per cent molybdenum steel are also presented in this paper.

This paper is a sequel to a paper presented before this Society ten years ago on a similar subject but embracing a material of different composition (**1**).[2] In 1941 central stations were operating nominally at 900 F with the highest temperature at 950 F. Present-day installations are being designed for nominal operating temperatures of 1000 and 1050 F with a high of 1100 F. The increase in central station superheat temperature of 100 to 150 F during the past decade has been accomplished in part through the development and use of materials having improved high-temperature properties, together with "know-how" in the manufacturing, handling, and fabrication of such materials. Over the past several years, 2¼ per cent chromium-1 per cent molybdenum (A 213 - 50 T, grade T22)[3] and 3 per cent chromium-1 per cent molybdenum steel (A 213 - 50 T, grade T21)[3] have been used for 1000 to 1050 F steam piping with the major tonnage being in the former grade. Since these two grades of material come within the classification of air-hardening steels, close procedure control must be exercised in fabrication.

Background in Use of Steam Piping Materials

In view of certain questions to be raised in this paper, it is desirable that a brief background be given of the se-

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* Presented at the Fifty-fourth Annual Meeting of the Society, June 18–22, 1951.

[1] Metallurgist, Pittsburgh Piping and Equipment Co., Pittsburgh, Pa.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 887.

[3] Specifications for Seamless Alloy-Steel Boiler and Superheater Tubes (A 213 - 50 T), 1950 Supplement to Book of ASTM Standards, Part 1, p. 14.

quence of events which has led to the use of the steam piping materials currently being specified. It may be safely stated that the development and use of low-carbon $\frac{1}{2}$ per cent molybdenum steel (ASTM Specification A 206 T)[4] was in part responsible for the increase in steam temperatures from approximately 825 to 950 F during the period 1934 to 1944. The failure of a large carbon molybdenum main steam pipe in 1943, however, was shown conclusively to be the result of high-temperature structural instability of this material. This failure resulted in a rude awakening to the fact that in addition to the slow mechanical plastic deformation (creep) which occurs with time at temperature, there also occurs a slow but simultaneous change in the metallurgical structure of such steels; in fact creep or any changes in creep rate are usually the result rather than the cause of changes in metallurgical structure. As a result of this failure and the many subsequent investigations of carbon-molybdenum steel pipe by the public utilities and other interested industrial laboratories, a preponderance of data was obtained which may be summed up as follows:

1. Carbon-molybdenum pipe deoxidized with little or no silicon and with aluminum in amounts of 1 to 2 lb per ton of steel was highly susceptible to structural decomposition and therefore not suitable for sustained temperatures of 950 F.

2. Carbon-molybdenum pipe, deoxidized principally with silicon and with little or no aluminum, was reasonably resistant to structural changes at 950 F although a sufficient number of exceptions were reported to seriously consider a more suitable alloy.

In August 1944, therefore, the first 1 per cent chromium-$\frac{1}{2}$ per cent molybdenum steel pipe (ASTM Specification A 213 - 50 T, grade T12)[3] was purchased for 950 F main steam line service. Restrictions on aluminum additions to the steel were imposed in accordance with the McQuaid-Ehn carburizing test which was subsequently incorporated in two ASTM pipe specifications (A 280 - 48 T[5] and A 315 - 48 T).[6] This material was intended as a replacement of, and a more suitable material than, carbon-molybdenum pipe for 950 F service. Within two years piping suitable for 1000 F was needed and designs for 1050 F steam generation had been accomplished. For these temperatures, a material higher in both chromium and molybdenum was needed for increased oxidation resistance and increased high-temperature strength, respectively. For this service the $2\frac{1}{4}$–3 per cent chromium, 1 per cent molybdenum steels have been used. Although the $2\frac{1}{4}$ per cent chromium, 1 per cent molybdenum steel has been quite generally accepted for 1000 F service, some differences of opinion appear to exist as to which material is preferable for 1050 F service. The differences appear to be whether a small known increase in high-temperature strength of the $2\frac{1}{4}$ per cent chromium, 1 per cent molybdenum alloy outweighs any possible benefits in the oxidation resistance of the 3 per cent chromium-1 per cent molybdenum alloy at 1050 F.

Since some grain refinement is accomplished by the addition of chromium to steel, the application of the McQuaid-Ehn test as specified in the lower chromium-molybdenum steels (ASTM Specifications A 280 - 48 T and A 315 - 48 T) is not too satisfactory when ordering 2¼ or 3 per cent chromium, 1 per cent molybdenum steel, since the results of such a test are not necessarily indicative of the use or extent of use of aluminum during the deoxidation process. The author is of the opinion, however, that the high-temperature properties of the intermediate chromium-molybdenum steels when melted to coarse grain melting practice (silicon killed) are superior to those when melted to fine grain melting practice (aluminum killed).

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[4] Standard Specifications for Seamless Carbon-Molybdenum Alloy-Steel Pipe for High Temperature Service (A 206 - 48 T), 1949 Book of ASTM Standards, Part 1, p. 159.

[5] Specifications for Seamless Chromium-Molybdenum Alloy-Steel Pipe for Service at High Temperatures (A 280 - 48 T), 1949 Book of ASTM Standards, Part 1, p. 278.

[6] Specifications for Seamless 1 Per Cent Chromium, 0.5 Per Cent Molybdenum Alloy-Steel Pipe for Service at High Temperatures (A 315 - 48 T), 1949 Book of ASTM Standards, Part 2, p. 295.

STEELS INVESTIGATED

The analyses and previous fabrication of the steels used in this investigation are given in Table I. Items Nos. 1, 2, 5, and 6 conform to the chemical requirements of ASTM Specification A 213 –50, grade T-22, with the exception that chromium in item No. 2 is three points under minimum. Item No. 3 conforms to the chemical requirements of ASTM Specification A 213 – 50, grade T-21, and item No. 4 conforms to the chemical requirements of ASTM Specification A 315 – 48 T. The latter is included principally for the purpose of contrasting certain mechanical and metallurgical properties of this material with those of the 2.25- and 3 per cent chromium, 1 per cent molybdenum grades. Items Nos. 7, 8, and 9 give the chemical analyses of weld deposits made with manual metal arc welding electrodes having different operational characteristics but suitable for the welding of 2¼ per cent chromium-1 per cent molybdenum steel piping.

TABLE I.—CHEMICAL ANALYSES AND PREVIOUS FABRICATION OF STEEL INVESTIGATED.

Item	Composition, per cent								Previous Fabrication
	Carbon	Manganese	Silicon	Phosphorus	Sulfur	Chromium	Molybdenum	Aluminum[a]	
No. 1......	0.15	0.46	0.27	0.020	0.012	2.26	1.00	None	10¾ by 1⅝-in. wall forged and bored pipe
No. 2......	0.13	0.36	0.015	0.005	0.021	1.97	1.16	...	8⅝ by 1-in. wall centrifugally cast pipe
No. 3......	0.09	0.45	0.26	0.014	0.030	3.10	0.88	...	11¾ by 1⅓-in. wall pierced and rolled pipe
No. 4......	0.13	0.54	0.22	0.020	0.027	0.83	0.50	0.44	10¾ by 1⅛-in. wall pierced and rolled pipe
No. 5......	0.12	0.43	0.27	0.011	0.020	2.24	0.98	None	10¾ by 1⁹⁄₁₆-in. wall pierced and rolled pipe
No. 6......	0.11	0.52	0.11	0.015	0.026	2.24	0.91	0.26	10¾ by 1⁹⁄₁₆-in. wall pierced and rolled pipe
No. 7......	0.10	0.40	0.28	...	...	2.35	1.08	...	E10010 weld metal
No. 8......	0.13	0.35	0.57	...	...	2.21	0.92	...	E10013 weld metal
No. 9......	0.10	0.53	0.49	...	...	2.36	0.96	...	E10016 weld metal

[a] Pounds per ton.

METALLURGICAL CHARACTERISTICS OF LOW-CARBON CHROMIUM-MOLYBDENUM STEEL PIPING

In discussing the metallurgical characteristics of high-temperature alloy-steel piping materials, it is of interest to know what changes in mechanical properties, if any, have taken place in the finished product as a result of the various cold or hot forming operations or from cooling from the temperatures employed in the hot fabricating operations. If changes have taken place in the material which make it unsuitable for service, the type of heat treatment required to restore the desired properties must be ascertained.

In determining the relative response of a given material to heat treatment, good use can be made of Jominy End-Quench hardenability data by comparing curves of a steel for which relatively little information is available with those of steels in which considerable fabricating data and background are available.

Such data for 2¼- and 3 per cent chromium, 1 per cent molybdenum steels are shown in Fig. 1 compared with those of the more commonly used 1 per cent chromium-$\frac{1}{2}$ per cent molybdenum material. From these data it may be noted that the maximum hardness of a given alloy when drastically cooled is principally a function of carbon content but the depth of hardness is principally a function of alloy content.

Although the chromium - molybdenum alloys are not water quenched in the interest of safe practice, they are in many cases allowed to air cool after hot fabrication. Since the values along the right of Fig. 1 represent hardness essentially from air-cooled specimens, it may be noted that a considerable difference in hardness may be expected between $2\frac{1}{4}$- and 3 per cent chromium, 1 per cent molybdenum alloys and those containing only 1 per cent chromium and $\frac{1}{2}$ per cent molybdenum, when air cooled from hot fabricating operations. An intermediate alloy steel having a hardness of 30 to 35 Rockwell C (290 to 330 Brinell) is inherently less ductile than one of a lower alloy content having a hardness of 10 Rockwell C (190 Brinell) and must be handled accordingly.

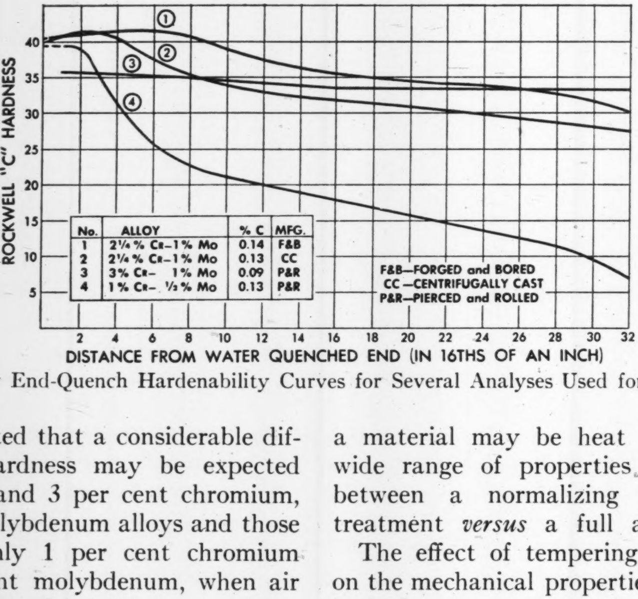


FIG. 1.—Jominy End-Quench Hardenability Curves for Several Analyses Used for Steam Piping

Since hardness of a material is related to microstructure and noting that the hardness of the water-quenched end of the $2\frac{1}{4}$- and 3 per cent chromium, 1 per cent molybdenum material is not materially different from that 2 in. from the water-quenched end (which approaches an air cool), it is not surprising therefore that the microstructure of this material when water quenched is similar to that when air cooled as shown in Fig. 2.

Slow controlled cooling of this alloy from 1600 F or above, however, does result in an entirely different microstructure with mechanical properties having widely different values from those obtained on air-cooled material.

With the difference in microstructure and corresponding hardness between the controlled slow-cooled (annealed) and air-cooled (normalized) specimens shown in Fig. 2, it is not surprising that such a material may be heat treated to a wide range of properties by choosing between a normalizing and temper treatment *versus* a full anneal.

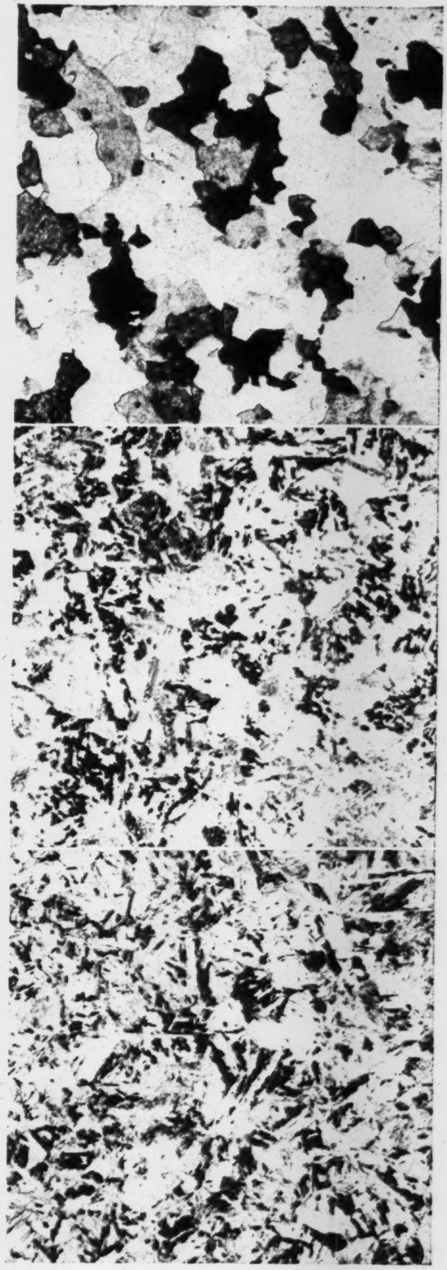
The effect of tempering temperature on the mechanical properties of normalized $2\frac{1}{4}$ per cent chromium, 1 per cent molybdenum steel is shown in Fig. 3. From these data it may be noted that this material may be heat treated to any tensile strength values ranging from 80,000 to 150,000 psi. From this figure it may also be observed that in order to avoid air hardening, a tempering temperature of 1400 F should not be exceeded unless adequate facilities are available for controlled slow cooling. It may also be noted from this figure that the material has passed through the transformation range at 1600 F and the mechanical properties again approach those of the material as originally normalized from 1750 F.

A similar curve to that of Fig. 3 is shown in Fig. 4 for a 3 per cent chromium, 1 per cent molybdenum material. It may be noted that the trend of these data is similar to that given in Fig. 3. The lower carbon content of this material is however reflected by somewhat lower hardness, tensile strength, and yield strength, and higher elongation and reduction of area for a given tempering temperature.

Although no use has been made of the process to date for high-pressure steam piping, considerable thought has been given to the use of centrifugally cast piping, and considerable research is currently under way. Economically this material offers advantage in that large quantities are not required to obtain rollings and the wall thickness can be varied as desired merely by controlling the quantity of metal poured into the mold. Comparing this process with bored forgings, the expense of hollow boring is also eliminated.

Shown in Fig. 5 is a comparison of data with Fig. 3 for a metal mold centrifugally cast $2\frac{1}{4}$ per cent chromium, 1 per cent molybdenum material. It is to be noted that the trend is similar and the comparison favorable to that of Fig. 3 although the hardness, tensile strength, and yield strength are low with respect to Fig. 3. This is no doubt due, however, to the lower carbon, manganese, silicon, and chromium values of the centrifugal casting with respect to the forged material. The values obtained for this material in the full annealed condition or normalized and tempered at 1300 to 1350 F condition are considered satisfactory in terms of specification requirements for similar wrought material.

Also of interest is the columnar or dendritic grain structure of the centrifugally cast material which results from



Top—Annealed, 155 Brinell
Middle—Normalized, 302 Brinell
Bottom—Water Quenched, 375 Brinell

FIG. 2.—The Effect of Cooling Rate from 1750 F on the Microstructure and Hardness of $2\frac{1}{4}$ per cent Chromium, 1 per cent Molybdenum Steel—Item No. 1 (× 100).

Etched in hydrochloric and picric acids.

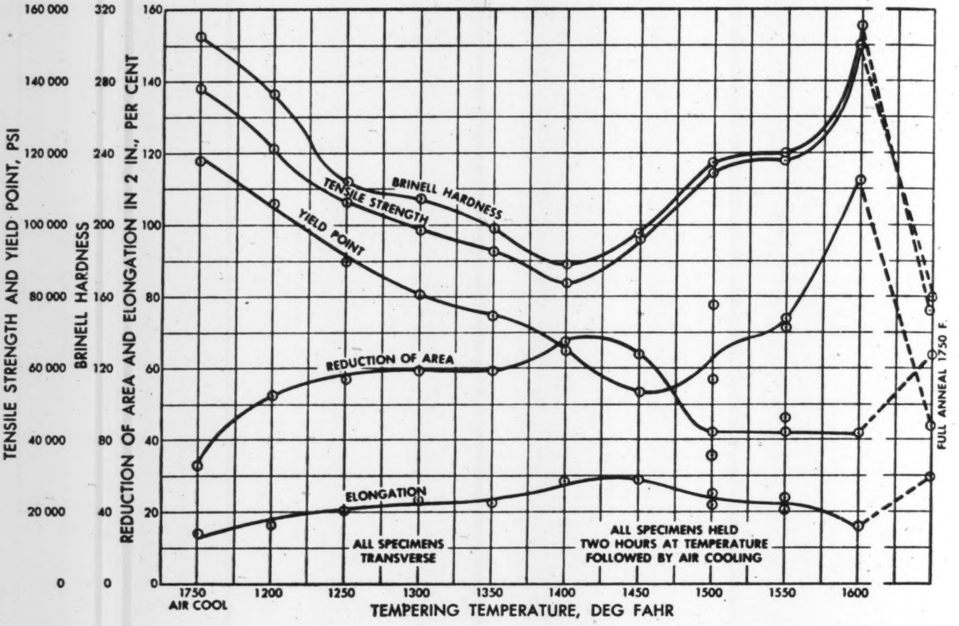


FIG. 3.—Effect of Tempering Temperature on Mechanical Properties of Forged and Bored $2\frac{1}{4}$ per cent Chromium, 1 per cent Molybdenum Steel Pipe Normalized From 1750 F—Item No. 1.

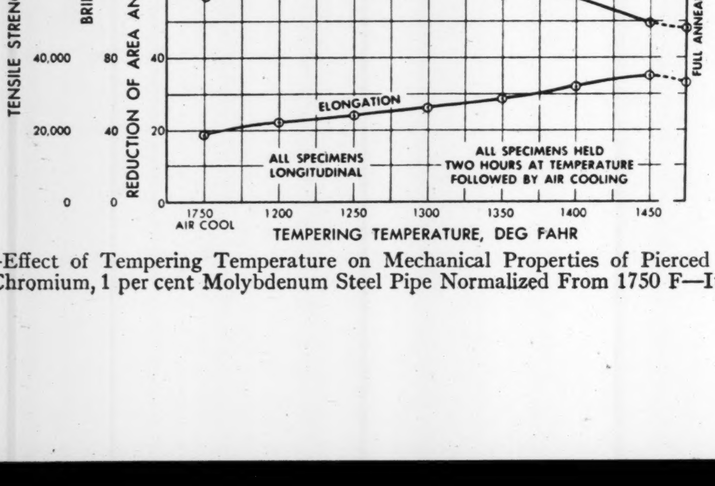


FIG. 4.—Effect of Tempering Temperature on Mechanical Properties of Pierced and Rolled 3 per cent Chromium, 1 per cent Molybdenum Steel Pipe Normalized From 1750 F—Item No. 3.

directional solidification from outside to inside diameter of the casting. This is shown in Fig. 6.

HOT FABRICATING OPERATIONS

The 2¼- and 3 per cent chromium, 1 per cent molybdenum alloys are not new materials and many fabricators have had some experience with these alloys for at least ten years. It has been only in the past two or three years, however, that fabrication of these materials has been required in pipe sizes of from 10 to 20-in. diameter and wall thicknesses of 1.5 to 3.5 in. Experience indicates that they may be successfully hot formed which includes hot bending, upsetting, swaging, forging, and welding. Likewise cold-forming operations can be accomplished to a limited extent on small diameter pipe. In general it may be stated that the hot and cold forming of these two alloys behave not too unlike those of lower alloy content with the

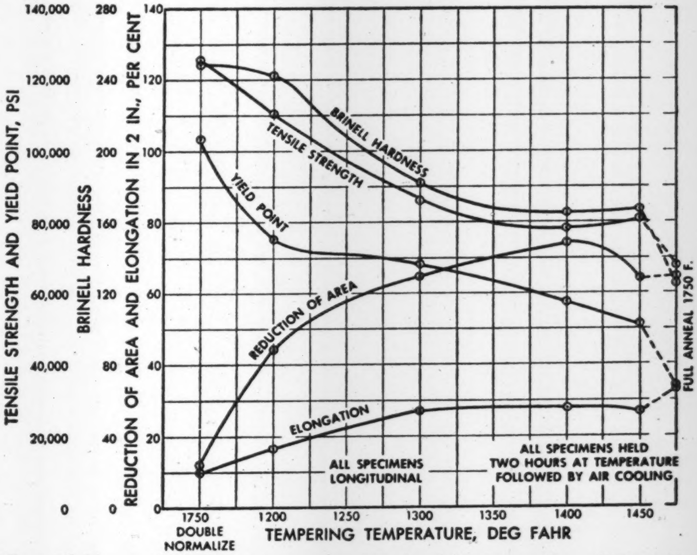


FIG. 5.—Effect of Tempering Temperature on Mechanical Properties of Centrifugally Cast 2¼ per cent Chromium, 1 per cnt Molybdenum Steel Pipe Double Normalized from 1750 F—Item No. 2.

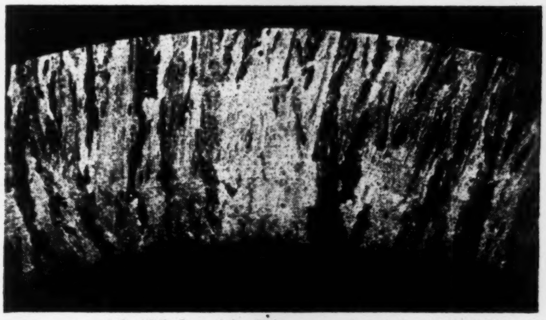


FIG. 6.—Macrostructure of 8.625 Outside Diameter by 1 in. Wall Centrifugally Cast 2¼ per cent Chromium, 1 per cent Molybdenum Steel Pipe Showing Columnar Grain Structure of "As Cast" Material (× 1).

Etched in 10 per cent nital.

exception of greater rigidity and therefore more power required per operation. Care must be exercised, however, in avoiding excessive strain on areas air cooled from previous hot fabricating operations.

HEAT TREATMENT FOR HIGH-TEMPERATURE STRENGTH

As most pipe fabricating operations are local in nature, the mechanical properties of fabricated pipe may vary considerably from one location in a pipe to another, with intervening areas having properties as acquired from the pipe mill. The purpose therefore of heat treating fabricated pipe is to produce a uniform grain structure throughout the entire length of each fabricated assembly with attendant uniform mechanical properties. Since service temperatures of 1000 to 1050 F on such piping assemblies are within the creep range of the material, the heat treatment applied should be that which will impart the most favorable creep or stress-rupture properties to the material. In so doing it must be kept in mind that the room temperature mechanical properties of any high-temperature alloy should provide for reasonable ductility and notch toughness so as to avoid the possibility of damage or fracture of the material during subsequent handling and erection.

While good judgement would preclude the possibility of overlooking the above, it likewise would preclude the possibility of heat treating for maximum softness and ductility at room temperature without regard for the strength of the material at the temperature for which it was designed to operate.

In addition to the above considerations, some knowledge of the notch toughness of the material at the operating temperature should be available.

The following questions therefore are raised:

1. What commercial heat treatment will impart the best high-temperature strength to $2\frac{1}{4}$ per cent chromium, 1 per cent molybdenum steel at 1000 F and at 1050 F? to 3 per cent chromium, 1 per cent molybdenum steel at 1050 F?
2. Does the structural grain size of these alloys have any material effect on their high-temperature strength?
3. Is a pearlitic type microstructure to be preferred to one of tempered martensite for good high-temperature properties at 1000 or 1050 F?
4. Does silicon killed $2\frac{1}{4}$- and 3 per cent chromium, 1 per cent molybdenum steel have better high-temperature strength than the same steel when deoxidized with aluminum, grain size and microstructure being comparable?

The best high-temperature properties for carbon steel, carbon-molybdenum, and low-chromium molybdenum steels (within the temperature range usually associated with the specific alloy for power plant designs) are obtained when the materials are silicon killed and, further, when such steels have a medium coarse grain size and Widmanstaetten carbide structure. Smith has presented an excellent discussion of these factors together with an extensive bibliography on this subject (**2**). It is recognized that the above outlined conditions would not necessarily apply to the specific alloy grades under discussion, nonetheless there does not appear to be sufficient data in the literature to refute the argument that the same factors which produce good high-temperature properties in the lower alloy steels would not do likewise in the $2\frac{1}{4}$ per cent chromium, 1 per cent molybdenum material.

Most of the high-temperature data available on the intermediate alloy grades under discussion are for material having a structurally fine grain size

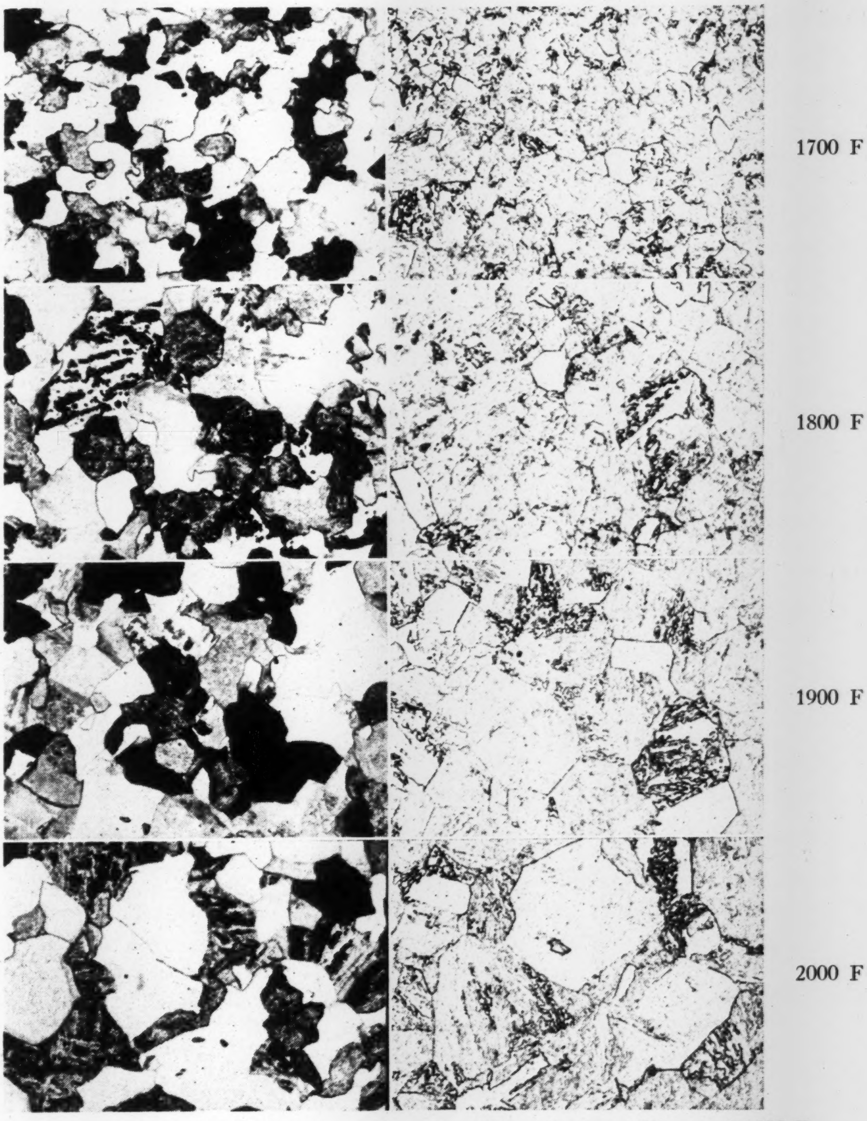

1700 F
1800 F
1900 F
2000 F
Annealed
Normalized and Tempered 1300 F

FIG. 7.—The Effect of Heat Treating Temperature and Mode of Cooling on the Structural Grain Size of $2\frac{1}{4}$ per cent Chromium, 1 per cent Molybdenum Steel—Item No. 1 (× 100).

Etched in hydrochloric and picric acids.

and pearlitic type of microstructure. A common heat treatment applied has been controlled slow cooling from temperatures of about 1575 F, a temperature which barely reaches the upper critical temperature.

In contrast to the extremely fine grain size (8 to 9, ASTM Classification of Austenite Grain Size in Steels E 19)[7] of material shown with established creep or stress-rupture values, the grain coarsening characteristics and microstructure of a silicon-killed $2\frac{1}{4}$ per cent chromium, 1 per cent molybdenum alloy is shown in Fig. 7.

It is suggested that a grain size (ASTM Nos. 4 to 6)[7] comparable to that obtained by heating to 1700 or 1800 F as shown in Fig. 7 would result in higher stress-rupture or creep values than would be obtained with similar material having an ASTM grain size of Nos. 8 to 9.

In support of the use of a coarse grain size, Thielemann obtained in a high-silicon, 3 per cent chromium, $\frac{1}{2}$ per cent molybdenum alloy, almost double the indicated 100,000 hr rupture strength at 1100 F of that obtained on the same alloy when in the fine grain condition (**3**).

Miller *et al* (**4**) obtained on normalized and tempered $2\frac{1}{4}$ per cent chromium, 1 per cent molybdenum steel a creep strength of 13,300 psi for a creep rate of 1 per cent in 10,000 hr which was 10 per cent higher than that obtained on the same material in the full annealed condition when tested at 1000 F.

With steel of the same nominal composition, Babcock & Wilcox (**5**) have reported 16,350 psi for a comparable creep rate and test temperature on full annealed material supposedly having a fine grain size. Noting that the silicon content of the steel tested by Babcock & Wilcox was 0.36 per cent as contrasted with 0.12 per cent in the steel tested by Miller, and in view of the recent uprating of the allowable design stress by the American Society of Mechanical Engineers' Boiler Code for the high-silicon $1\frac{1}{4}$ per cent chromium, 0.5 per cent molybdenum material (ASTM Specification A 158 - 50 T, grade P11)[8] to an identical value at 1000 F of that assigned to the $2\frac{1}{4}$ per cent chromium grade which has a higher molybdenum content, the question is raised as to the advisability of specifying a silicon range of 0.25 to 0.50 per cent rather than 0.50 per cent maximum, as found in current specifications.

It is believed that certain of the original applications for $2\frac{1}{4}$- and 3 per cent chromium, 1 per cent molybdenum material was in boiler superheater tubes and oil cracking furnaces where the temperature might frequently or constantly be at or above 1100 F, and under such circumstances full annealing for maximum softness would be desirable.

A carryover of this practice in the application of this material to high-temperature steam piping for 1000 or 1050 F service may be correct, but not necessarily so.

When heat treating material for high-temperature service, it is believed that the heat treatment should be such that a reasonable stability of microstructure will be maintained at the service temperature.

STRUCTURAL STABILITY OF $2\frac{1}{4}$ PER CENT CHROMIUM-1 PER CENT MOLYBDENUM STEEL AT 1050 AND 1150 F

In an endeavor to determine the microstructural stability of $2\frac{1}{4}$ per cent chromium, 1 per cent molybdenum steel as a function of hardness, three melts of this material were selected. Material from each melt was divided into two lots, one of which was given a 1650 F full anneal, and the other given a 1750 F normalizing treatment and 1300 F temper heat treatment. Each of the six lots of material was further divided into three groups. Hardness and Charpy impact tests were made on one group in the "as-heat-treated" condition, and the other two groups were aged for 2000 hr at 1050 F and 1150 F, respectively. Hardness readings were taken at the end of approximately 100, 500, 1000, and 2000 hr. Impact tests were then made at 1050 and 1150 F on the balance of the aged material. The effect of aging time on the Brinell hardness of the three melts, in two conditions of heat treatment when aged at both 1050 and 1150 F is shown plotted in Fig. 8. The results of the Charpy impact tests made at room temperature prior to aging, as well as those made at 1050 and 1150 F subsequent to 2000 hr aging at the respective temperatures are given in Table II. Shown in Figs. 9 and 10, is the microstructure of the full-annealed, and normalized-and-tempered material respectively, both before and after aging at 1150 F. As no difference could be detected in the microstructure of two melts (items 1 and 5), photomicrographs from only two melts (items 5 and 6) are presented. From Fig. 8 it may be observed that at 1150 F a rapid and distinct loss in hardness occurred in all normalized-and-tempered specimens as well as some loss in hardness of two of the annealed specimens. On the basis of microstructural stability therefore, this material, if subjected to operating temperatures at or near 1150 F, should definitely be given a full anneal. The loss in hardness at 1150 F of both items 1 and 5 in the annealed condition indicates that the cooling rate of these two melts should have been even slower than that given during the original annealing treatment. The low-carbon, low-silicon melt only, in the annealed condition, is representative of a stable microstructure.

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[7] 1949 Book of ASTM Standards, Part 1, p. 1271.

[8] Tentative Specifications for Seamless Alloy-Steel Pipe for High-Temperature Service (A 158 - 50 T), 1950 Supplement to Book of ASTM Standards, Part 1.

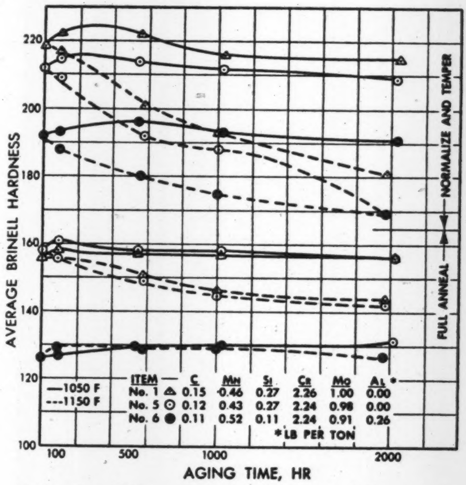


FIG. 8.—Effect of Aging Time and Temperature on The Brinell Hardness of Full Annealed and Normalized and Tempered 2¼ per cent Chromium, 1 per cent Molybdenum Steel.

TABLE II.—TRANSVERSE CHARPY IMPACT TESTS MADE ON 2¼ Cr-1 Mo STEEL.[a]

Item	Average Impact Values					
	70 F		1050 F[b]		1150 F[b]	
1650 F ANNEALED						
No. 1	33.5	32.2	35.5	34.5	35.5	35.0
No. 1	31.0		33.5		34.5	
No. 5	34.0	31.5	35.5	34.7	33.0	37.5
No. 5	29.0		34.0		42.0	
No. 6	37.5	35.5	30.0	30.0	27.0	28.5
No. 6	33.5		30.0		30.0	
1750 F NORMALIZE—1300 F TEMPER						
No. 1	33.5	36.2	30.0	29.0	31.0	30.5
No. 1	39 0		28 0		30 0	
No. 5	39.5	36.5	27.5	27.5	31.5	30.7
No. 5	33.5		27.5		30.0	
No. 6	38.0	39.5	21.5	22.0	20.0	23.2
No. 6	41.0		22.5		26.5	

[a] Specimen type C, ASTM Standard Methods of Impact Testing of Metallic Materials, E 23 - 47, 1949 Book of ASTM Standards, Part 1, p. 1287, Part 2, p. 1035.

[b] Specimens aged 2000 hr temperature prior to testing.

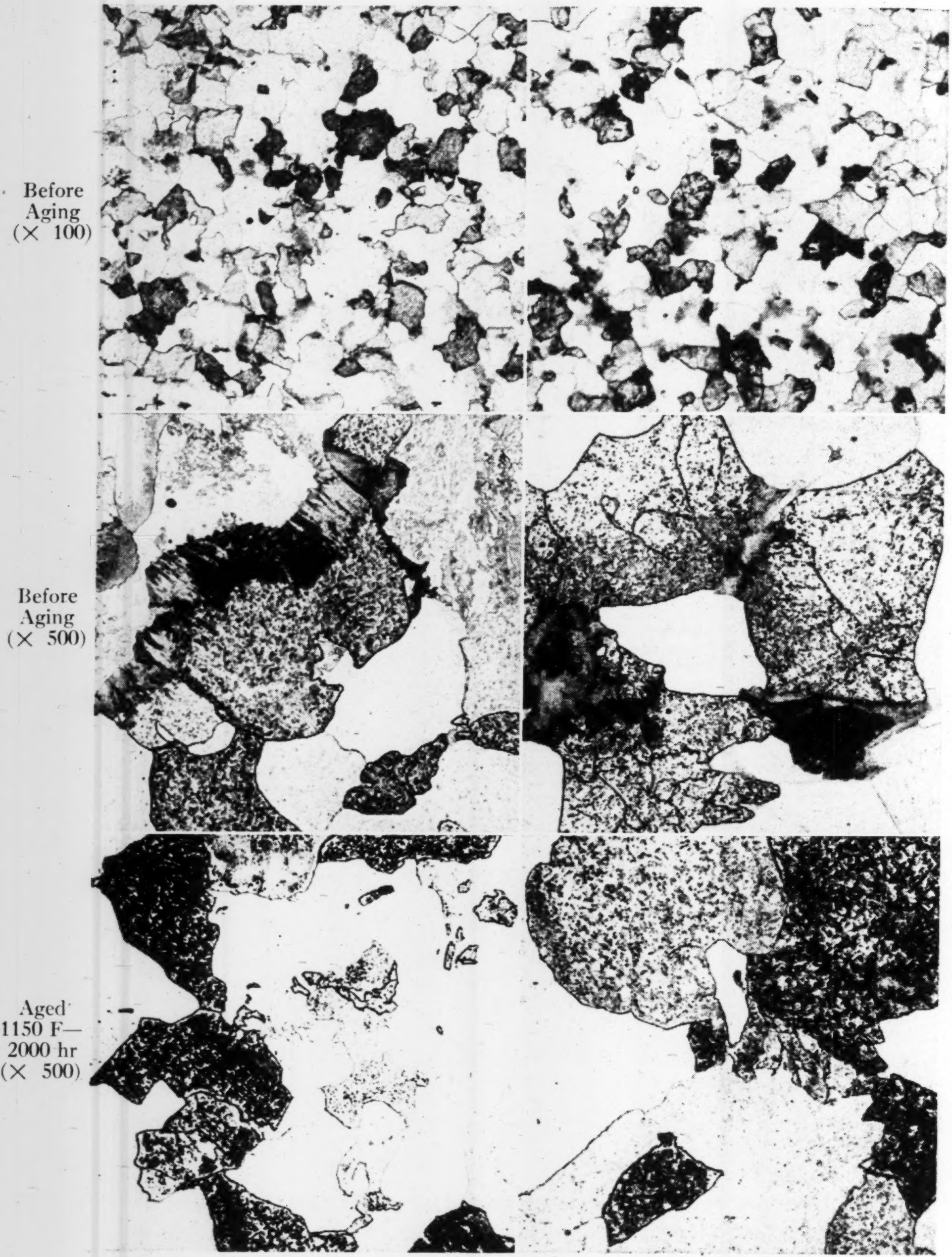
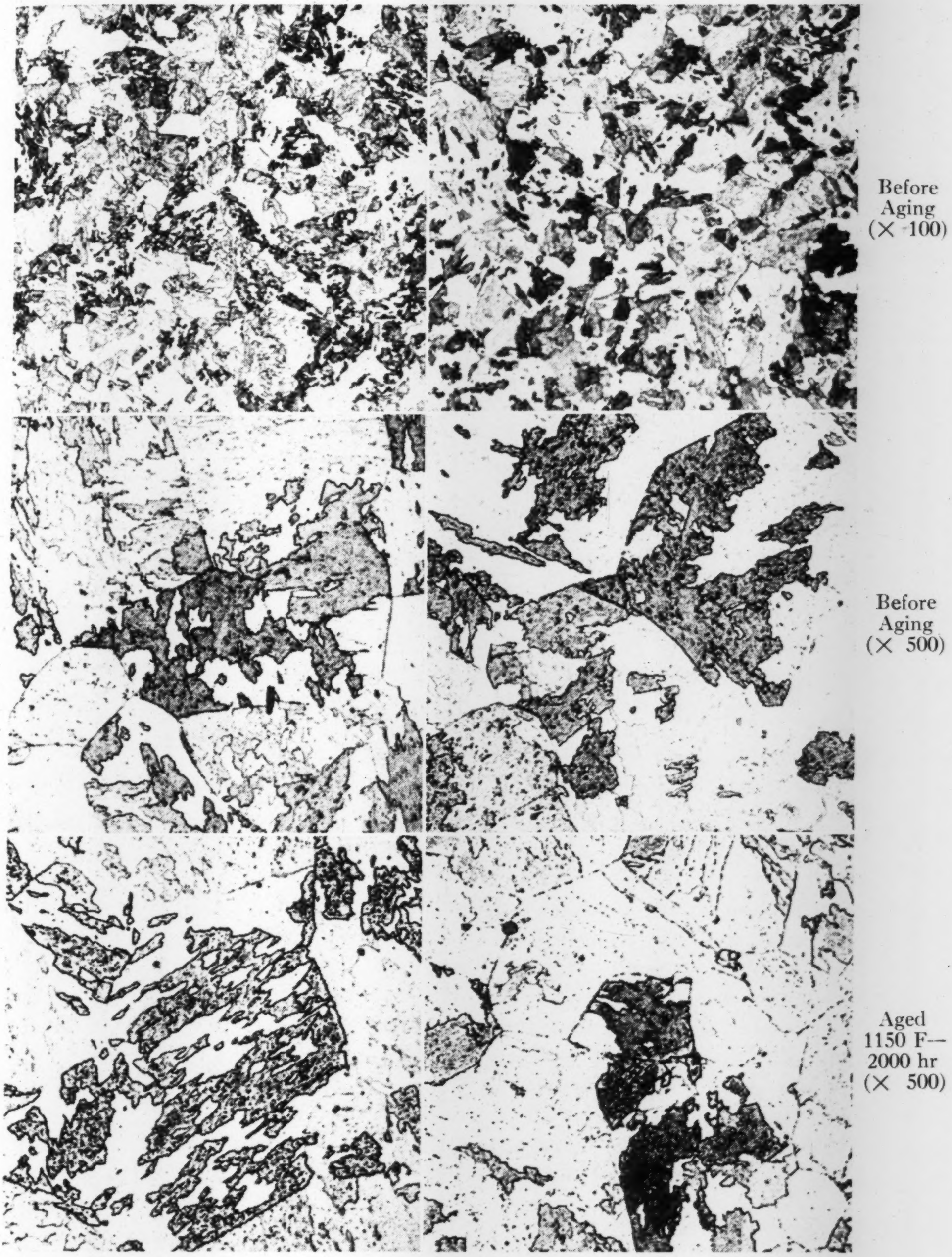

Before
Aging
(× 100)
Before
Aging
(× 500)
Aged
1150 F—
2000 hr
(× 500)
Item No. 5
Item No. 6

FIG. 9.—Microstructures of Annealed 2¼ per cent Chromium, 1 per cent Molybdenum Steel Before and After Aging at 1150 F for 2000 hr.

Etched in hydrochloric and picric acids.


Before Aging (× 100)
Before Aging (× 500)
Aged 1150 F— 2000 hr (× 500)

Item No. 5 Item No. 6

FIG. 10.—Microstructures of Normalized and Tempered 2¼ per cent Chromium, 1 per cent Molybdenum Steel Before and After Aging at 1150 F for 2000 hr.

Etched in hydrochloric and picric acids

At 1050 F, however, it is to be noted that a reasonable stability of microstructure exists in all specimens in both conditions of heat treatment as evidenced by the fact that the hardness of each specimen after 2000 hr is within 1 to 4 Brinell hardness points of that obtained prior to aging.

It is to be further noted that at 1050 F mild precipitation hardening appears to have taken place, with maximum hardness occurring between 100 and 500 hr with the exception of the annealed low-carbon low-silicon alloy, which exhibits slight but highest hardness after 2000 hr at temperature. Confirmation of the increase in hardness at 1050 F and decrease in hardness at 1150 F is given in the publication by Miller *et al* (4) who found an increase in hardness from 159 to 163 to 167 Brinell after creep testing normalized-and-tempered $2\frac{1}{4}$ per cent chromium-1 per cent molybdenum steel at 1000 F for 3000 hr. The same material in the annealed condition and under the same conditions of test increased in hardness from 133 to 136 Brinell. When testing this material in creep at 1100 F in the normalized-and-tempered condition, however, a decrease in Brinell hardness from 159 to 136 was observed after 3000 hr. From these data as well as those shown in Fig. 8 it appears clear that for reasonable microstructure stability at temperatures above 1050 F, full annealing is preferable to a normalizing and tempering treatment, whereas, at a temperature of 1000 F or lower, microstructural stability is assured by either method of heat treatment. Although the original hardness was essentially maintained at 1050 F for 2000 hr in the normalized-and-tempered material, whether any substantial drop would occur over 100,000 hr at temperature cannot be definitely stated.

Little or no difference could be observed in the microstructure of the annealed specimens before and after 2000 hr at 1150 F other than a very slight increase in the amount of carbide precipitation after 2000 hr at temperature. Carbide precipitation was more noticeable however in the normalized-and-tempered specimens after 2000 hr at 1150 F, particularly item No. 6.

From Table II it may be noted that transverse Charpy impact tests made at room temperature were higher in the normalized-and-tempered condition than in the annealed condition, but when made at a temperature of 1050 and 1150 F, the reverse was true. Comparing the tests made at 1050 F with those made at 1150 F, the average test values at 1150 F were, in general, slightly higher than those made at 1050 F. It is difficult to state whether the slight increase was due to the increase in test temperature or the result of a lower hardness of the 1150 F specimens compared with the 1050 F specimens as a result of the 2000 hr aging treatment prior to making the impact tests. Comparing the tests made at 70 F with those made at elevated temperature, it may be observed that the test values obtained on the normalized-and-tempered specimens at 70 F were in all cases higher than those obtained at 1050 or 1150 F, whereas in two out of three melts, the reverse was true in the annealed specimens. Although the impact values are not high, it is believed that they represent a reasonable margin of safety insofar as concerns notch toughness of this material at both room temperature and at the service temperature.

THE MECHANICAL AND METALLURGICAL PROPERTIES OF WELDED JOINTS

The sequence of operations in the fabrication of $2\frac{1}{4}$ per cent chromium-1 per cent molybdenum piping is essentially that used in the fabrication of carbon-molybdenum piping a decade ago. After all the hot and cold working operations have been completed, the pipe is heat treated for uniformity of grain size and mechanical properties. The pipe ends are subsequently machine beveled and fitted up for welding.

Welding practice consists of preheating to 500 to 600 F and maintaining this temperature during welding, followed by stress relieving, perhaps more properly termed a post welding heat treatment, the terminology depending somewhat on the temperature to which the joint is heated after welding. Some differences of opinion appear to exist as to the proper post weld heating temperature, as variations exist from 1300 to 1375 F with, in some instances, temperatures as high as 1525 F being used.

This variation in temperature is believed to be the result of an attempt to obtain 30 per cent stretch in the weld deposit by free-bending as called for by the ASME Boiler Construction Code. The validity of this requirement for weld deposits regardless of chemical composition is seriously questioned, and in striving for this arbitrary room temperature requirement various stress-relieving temperatures have been found to be required depending principally upon the choice of the welding electrode and the mechanical properties of the base metal being welded.

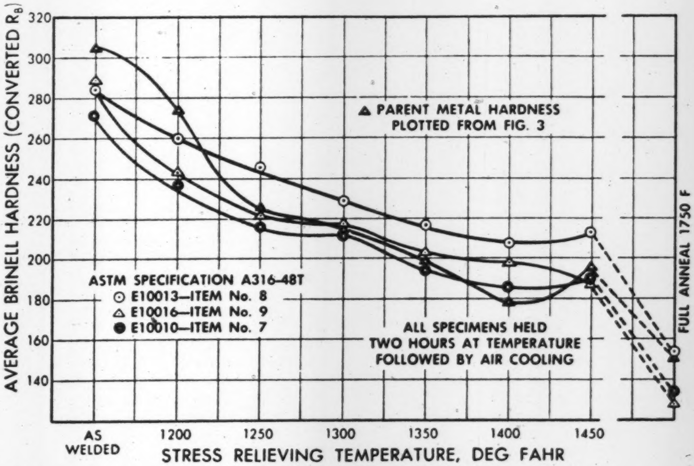


FIG. 11.—Effect of Stress Relieving Temperature on Brinell Hardness of $2\frac{1}{4}$ per cent Chromium, 1 per cent Molybdenum Welds Deposited With Electrodes Having Variations in Operational Characteristics.

Plotted in Fig. 11, as a function of stress relieving temperature, is the hardness of multiple bead weld deposits made with three $2\frac{1}{4}$ per cent chromium-1 per cent molybdenum electrodes having different operational characteristics. Superimposed on this plot is the parent metal hardness taken from Fig. 3. While all three electrodes are capable of making a satisfactory weld, it is obvious that a weld deposited with the E10013 electrode would be the most difficult to obtain a given weld metal elongation since the hardness and therefore yield and tensile strength is greater than the base metal at all stress-relieving temperatures above 1225 F. When such a condition exists, a large percentage of the stretch occurs in the softer parent metal adjacent to the weld. A similar but exaggerated condition frequently occurs as the result of making a welding procedure test on pipe which has been furnished from the mill in the annealed condition. From Fig. 11 it may be observed that the hardness of the base metal in the annealed condition is 151 Brinell and that of the E10016 weld deposit 203 Brinell when stress relieved at 1350 F. The difference in hardness between the weld deposit and weld-heat-affected zone of the base metal and that of the unaffected adjacent annealed parent metal is so great that essentially all the stretch occurs in the base metal adjacent to the weld. Such a test does not disprove the adequacy of the electrode nor of the welding procedure but merely proves the inadequacy of the base metal to force the weld metal to elongate. To obtain the required stretch in weld metal under such conditions necessitates a considerably higher post weld heating temperature with controlled slow cooling in order to soften the weld deposit to a hardness comparable to that of the base metal.

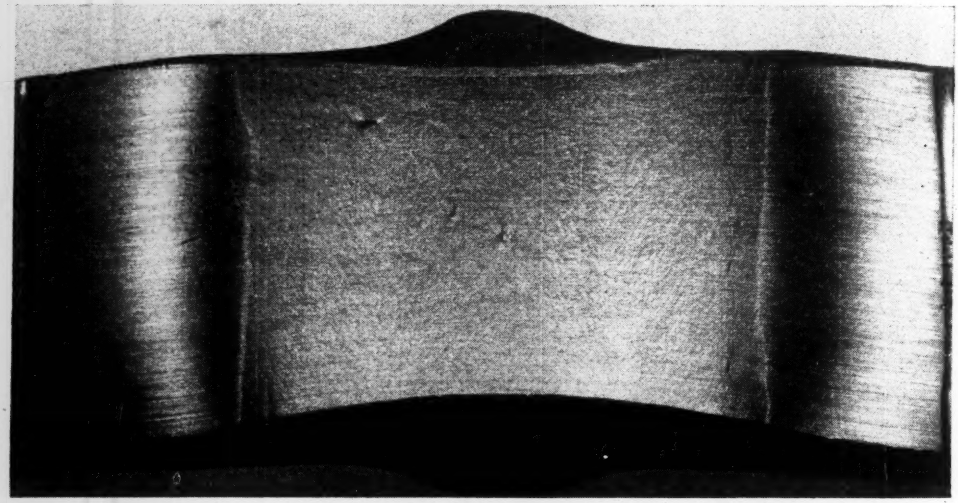


FIG. 12.—Specimen Cut From $10\frac{3}{4}$-in. Outside Diameter by $1\frac{9}{16}$-in. Wall Welded Joint in $2\frac{1}{4}$ per cent Chromium, 1 per cent Molybdenum Steel Pipe Stress Relieved at 1300 F (× 1).

60 per cent elongation across weld face after bending.

From Fig. 11 it may be noted that the hardness of an E10016 weld deposit stress relieved at 1300 F is essentially the same (216 Brinell) as that of the normalized parent metal when tempered at 1300 F. A bend specimen cut from a weld test made under the above conditions is shown in Fig. 12. Forced bending through the weld area resulted in a weld metal elongation of 60 per cent.

Although originally three types of electrodes were available for welding $2\frac{1}{4}$ per cent chromium, 1 per cent molybdenum steel, typical analyses for which are given in Table I, the use of the low-hydrogen E10016 electrode is presently considered preferable. While excellent mechanical properties can be obtained from weld deposits made with this electrode, it is unquestionably the most critical electrode from the standpoint of operational characteristics. The electrode coating is hygroscopic and sensitive to moisture pick-up. Weld deposits made with excessive moisture in the coating will result in considerable porosity in the deposit. A relatively high welding current and short arc is further required to prevent porosity. Porosity is also frequently found at stops and starts, thus making the overhead position of fixed horizontal welds especially prone toward porosity.

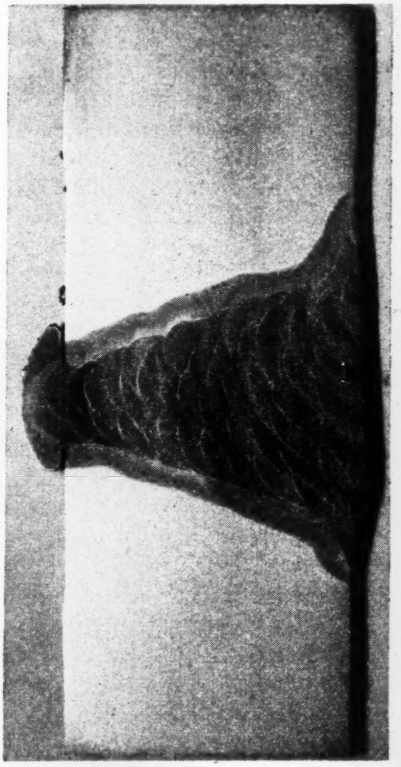


FIG. 13.—Fixed Vertical Pipe Weld. Electrode E10013 (×1).

Etched in 10 per cent nital.

The sequence of weld beads in a $1\frac{9}{16}$-in. wall fixed vertical and fixed horizontal pipe joint is shown respectively in Figs. 13 and 14. The cross-sections shown in these figures are characteristic of those found in heavy wall pipe joints used for high-temperature service.

Given in Table III is the range in mechanical properties to be expected in welded and stress-relieved pipe joints of a $2\frac{1}{4}$ per cent chromium, 1 per cent molybdenum analysis.

SUMMARY

The intermediate alloy steels of the $2\frac{1}{4}$- and 3 per cent chromium, 1 per cent molybdenum analyses are unlike those of lower alloy content in that they approach full hardness upon air cooling from hot fabricating operations when temperatures of 1600 F or higher are

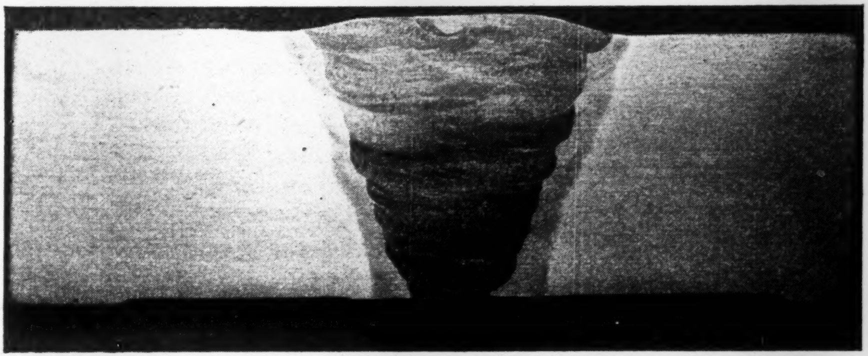


FIG. 14.—Fixed Horizontal Pipe Weld. Electrode E10016 (× 1).

Etched in hot 50 per cent hydrochloric acid.

employed—in fact, 2¼ per cent chromium, 1 per cent molybdenum annealed material will increase in hardness and strength with some loss in ductility when air cooled from a temperature as low as 1450 F. A corresponding temperature for the 3 per cent chromium, 1 per cent molybdenum grade is 1500 F.

Although no use has been made of the process to date for high-temperature steam piping, considerable thought has been given to the use of centrifugally cast alloy steel piping. Preliminary data presented indicates the properties of this material to be favorable to those of a wrought product.

TABLE III.—MECHANICAL PROPERTIES OF WELDED JOINTS IN 2¼ Cr-1 Mo PIPE AFTER STRESS RELIEVING AT 1300 F TO 1400 F.

Reduced Section Tensile Strength, psi	Bend Ductility Elongation in Outside Fibers, per cent	Brinell Hardness
65 000 to 95 000 (failure in parent metal).......	30 to 60	185 to 230

When heat treating 2¼- or 3 per cent chromium, 1 per cent molybdenum material for subsequent service at 1000 or 1050 F, the following questions are raised:

1. Does the structural grain size materially affect the high-temperature creep or the stress-rupture properties of such alloys?
2. At 1000 or 1050 F, is there any significant difference between the creep or stress-rupture properties of annealed *versus* normalized and tempered materials?
3. Are the creep or stress-rupture properties of 2¼- and 3 per cent chromium, 1 per cent molybdenum steels as sensitive as carbon steel, carbon-molybdenum, and low-chromium-molybdenum steels are to aluminum *versus* silicon deoxidation?

Aging tests conducted at 1050 and 1150 F for 2000 hr revealed:

1. The occurrence of mild precipitation hardening followed by slight softening of both annealed and normalized-and-tempered specimens at 1050 F.
2. Good structural stability of both annealed and normalized-and-tempered specimens at 1050 F.
3. Superior structural stability of material in the annealed condition at 1150 F.

Impact tests conducted at both room temperature and 1050 and 1150 F revealed:

1. The room temperature impact values to be higher in the normalized-and-tempered condition than in the annealed condition.
2. The high-temperature impact values to be higher in the annealed condition than in the normalized-and-tempered condition.
3. The impact test values obtained at 1150 F to be slightly higher than those obtained at 1050 F.
4. The impact values at elevated temperatures were lower than those at room temperature in the normalized-and-tempered condition, whereas the reverse was true in the annealed specimens.

It is believed however that the impact values obtained at both room temperature and elevated temperature represent a reasonable margin of safety insofar as notch toughness of this material is concerned.

Welding practices are at this time reasonably well standardized except perhaps for the post weld heating temperature which may vary from 1300 F to as high as 1525 F depending principally upon ability to obtain an arbitrary value of 30 per cent stretch across the face of a weld deposit, which is governed largely by the type of electrode used and the mechanical properties of the base metal being welded. Satisfactory welds can be made in heavy wall (1.5 to 3.5 in.) $2\frac{1}{4}$- and 3 per cent chromium, 1 per cent molybdenum piping having mechanical properties equal to that of the parent metal.

Acknowledgements:

The author wishes to acknowledge the assistance of Matthew Morrow, Wendell Hutchinson, and William Gilson in carrying out the tests conducted. The author is indebted to the United States Pipe and Foundry Co. for their part in furnishing the centrifugally cast piping.

REFERENCES

(1) R. W. Emerson, "The Fabrication of Carbon-Molybdenum Piping for High Temperature Service," *Proceedings*, Am. Soc. Testing Mats., Vol. 41, p. 579 (1941).

(2) George V. Smith, "Properties of Metals at Elevated Temperatures," The McGraw-Hill Book Co., Inc., New York, N. Y. (1950).

(3) R. H. Thielemann, "Some Effects of Composition and Heat Treatment on the High Temperature Rupture Properties of Ferrous Alloys," *Proceedings*, Am. Soc. Testing Mats., Vol. 40, p. 788 (1940).

(4) R. F. Miller, W. G. Benz and M. J. Day, "Creep Strength, Stability of Microstructure and Oxidation Resistance of Cr-Mo and 18 Cr-8 Ni Steels," *Transactions*, Am. Soc. Metals, Vol. 32, p. 381 (1944).

(5) "Properties of Carbon and Alloy Seamless Steel Tubing for High-Temperature and High-Pressure Service," *Technical Bulletin No. 6-E*, Babcock and Wilcox Tube Co. (1948).

DISCUSSION

MR. J. J. KANTER.[1]—There is one question Mr. Emerson raises that I should like to comment on particularly. Quoting from his paper:

"When heat treating $2\frac{1}{4}$ per cent chromium, 1 per cent molybdenum material for subsequent service at 1000 or 1050 F, the following questions are raised:

"Does the structural grain size materially affect the high-temperature creep and the stress rupture properties of such alloys?"

I think it very definitely does. Creep stress values from three different manufacturers of this alloy, presumed to be fine-grained material, are shown below:

CREEP TEST DATA FOR 2¼ CHROMIUM-1 MOLYBDENUM STEEL AT 1050 F.

Material	ASTM Grain Size No.		Creep Stress, psi	
	Mc-Quaid-Ehn Test	Ferrite	1% Rate per 10^5 hr	1% Rate per 10^4 hr
Tubing A	[a]		5000	8 100
Tubing B	[a]		5600	9 800
Tubing C	[a]		5800	12 200
Forged and bored pipe, 3¼-in. wall	1 to 4	4 to 5	7900	11 300
Forged and bored pipe, 4-in. wall	1 to 4	0	9000	13 200

[a] Presumably fine grain steel (No. 6 to 8 grain size).

Recently, the A.S.M.E. Boiler Code Committee, being guided primarily by this information, set an allowable stress value for this steel at 1050 F of 5800 psi.

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[1] Directing Engineer, Engineering Laboratories, Crane Co., Chicago, Ill.

At Crane Co. we have had an opportunity to study some of the coarser grain materials in this composition which were obtained from hollow bored pipes of 3¼-in. and 4-in. wall thickness for a 1050 F plant.

Creep stress data have been obtained at 1050 F in two series of tests on these steels. One had a grain size of No. 4 to 5 and, for a rate of 1 per cent per 100,000 hr, gave a stress value of 7900 psi. The other steel of exceptionally coarse grain (No. 0 and larger) yielded a value of 9000 psi. On the strength of this information, it seems there can be no question but that coarse grain practice produces better creep resistance in this composition. Additional creep and rupture data are being obtained for these steels.

It might be noted that end-quench hardenability curves for these steels are quite similar to those presented by Mr. Emerson. Also, structural stability at elevated temperatures is confirmed too by room temperature Charpy impact values of 38 and 33 ft-lb after 10,000 hr exposure at 1000 F and 1100 F, respectively.

MR. J. T. MACKENZIE.[2]—I would like to ask the author whether the centrifugal tube was cast on a sand or metal mold, and how much clean-up was taken out of the bore.

MR. R. W. EMERSON (*author*).—The centrifugally cast tube was cast in a metal mold. The tube was turned on the outside diameter and bored on the inside diameter, when received by the author. The actual amount of metal removed by machining is not known.

MR. A. B. WILDER.[3]—Reference was made to centrifugally cast seamless pipe. Another type of material which has recently become available is a seamless pipe manufactured by the cup and draw process from plate material. Mr. Emerson has had experience in the fabrication of this type of material. The mechanical properties, at ambient temperature of cupped and drawn pipe are quite similar to the rotary pierced Mannesman product. We also expect the high-temperature strength properties to be similar to the Mannesman product.

We have in progress exposure tests for 100,000 hr at 900, 1050, and 1200 F on the types of steels discussed by Mr. Emerson. In the 1951 *Transactions* of the American Society for Metals,[4] test data for the ½ chromium, ½ molybdenum and 3 chromium, 1 molybdenum steels are presented after 10,000 hr exposure at 900 and 1050 F.

In dealing with the types of steel discussed by Mr. Emerson, we found they are relatively stable at the high temperatures with respect to ordinary mechanical properties. The hardness, Charpy impact strength and tensile properties are reasonably stable after 10,000 hr exposure in our tests. After 34,000 hr exposure we have examined only the microstructure and find there is no appreciable change. We have found no graphite in any of the specimens, including the weld specimens.

I have data to present on ½ per cent chromium, ½ per cent molybdenum steel which had a carburized austenitic grain size of No. 1 to 3, and the 3 per cent chromium, 1 per cent molybdenum steel which had a carburized austenitic grain size of No. 5 to 7. The ½ per cent chromium, ½ per cent molybdenum steel was melted to coarse-grained deoxidation practice, and the 3 per cent chromium, 1 per cent molybdenum steel to fine-grained deoxidation practice. The ½ per cent chromium, ½ per cent molybdenum steel was in normalized condition (1650 F) and the 3 per cent chromium, 1 per cent molybdenum steel was in the annealed condition (1550 F).

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[2] Technical Director, Am. Cast Iron Pipe Co., Birmingham, Ala.

[3] Chief Metallurgist, National Tube Co., United States Steel Corp. Subsidiary, Pittsburgh, Pa.

[4] A. B. Wilder and J. O. Light, "Long-Time Elevated Temperature Test of Chromium-Molybdenum Steels, *Transactions*, Am. Soc. Metals, Vol. XLM, p. 323 (1951).

The creep rupture properties before and after 10,000 hr exposure are as follows:

	Stress For Rupture in 10,000 hr, psi			
	900 F		1050 F	
	Exposed	Unexposed	Exposed	Unexposed
½ per cent chromium, ½ per cent molybdenum	53 500	59 000	11 200	20 000
3 per cent chromium, 1 per cent molybdenum	27 500	29 000	10 500	11 300

It is rather significant to point out the fact that the 3 chromium, 1 molybdenum has creep rupture values considerably lower than the $\frac{1}{2}$ chromium, $\frac{1}{2}$ molybdenum steel, but after exposure at 1050 F for 10,000 hr, they are quite similar. I think this emphasizes what Mr. Emerson and Mr. Kanter have brought out, that is, heat treatment and other factors have a profound influence on the behavior of chromium-molybdenum steels under the conditions of creep rupture.

MR. H. D. NEWELL[5] (*by letter*).—Mr. Emerson's paper will be of interest to many piping fabricators and users of the 2 to 3 per cent chromium-molybdenum alloy steels which have recently come into use for Central Station steam piping for steam at 1000 to 1050 F. As the originator of the 3 chromium, 1 molybdenum steel[6] and the $2\frac{1}{4}$ chromium, 1 molybdenum steel[6], it may be of interest to supply some information relating to the development of these two popular high-temperature steels. The facts relating to this matter are:

The 3 chromium, 1 molybdenum steel was suggested, after due consideration, for a thermal polymerization-reforming oil refinery operation when the then-existing chromium-molybdenum (1936) steels were considered to be more than adequate for the corrosion to be encountered in the process but without adequate creep or rupture strength properties for the pressures involved. The 5 chromium, $\frac{1}{2}$ molybdenum steel, a popular oil refinery steel, was considered inadequate from a strength standpoint but more than sufficiently corrosion resistant, whereas a 2 chromium, $\frac{1}{2}$ molybdenum steel was perhaps barely corrosion resistant enough but lacked a desirable margin of strength properties where the product temperature was expected to be 1050 to 1070 F with metal temperatures perhaps approaching 1150 F on the furnace tubing with operating pressures ranging from 1600 to 2200 psi. After furnishing a quantity of the new 3 chromium, 1 molybdenum alloy steel tubing for an installation, further development work was then undertaken to determine the effect on creep strength properties on modifying the chromium content downward to $2\frac{1}{2}$ per cent and 2 per cent respectively, both with 1 per cent molybdenum. Creep tests on these experimental steels indicated they were nearly identical in creep strength within this chromium range and thereupon the average chromium content was selected as the nominal content for the second alloy, which alloy now corresponds to grade T 22. These steels had slightly improved creep strength properties over the original 3 chromium, 1 molybdenum steel and perhaps for that reason the $2\frac{1}{4}$ chromium, 1 molybdenum steel has become somewhat more popular in general use.

Following expanded use in the oil refining industry, these steels were next adapted for steam superheater tubes where they are now widely employed to a limiting metal temperature of about 1100 F. Refinery heater tubing, hot oil piping, and superheater tubes are required to be sufficiently soft and ductile for expanding into headers or for bending and other fabricating procedures, and since relatively high operating temperatures were expected in any case, it became general practice to furnish these alloy materials in the fully annealed condition and since it was felt, also, that a heat-treated material would ultimately soften in service. Present specifications such as A 200 - 51 T for alloy steel still tubes, and A 213 - 52 T[7] for seamless alloy steel boiler, superheater, and heat exchanger tubes include these alloy steels. These specifications contain elongation, deformation, and hardness test requirements most readily met by material in the fully annealed condition, although normalizing and long-time tempering has sometimes been used as an alternate treatment. These two grades are contained also in the new tentative Specifications for Seamless Ferritic Alloy Steel Pipe for High-Temperature Service, A 335 - 51 T.[7]

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[5] Chief Metallurgist, The Babcock and Wilcox Tube Co., Beaver Falls, Pa.

[6] U. S. Patent No. 2,182,177.

The first fifty or more electric furnace heats of these steels for tubing were made with straight silicon deoxidation, without aluminum additions on the premise that they would be more creep resistant if the aluminum was omitted. They are relatively through-hardening, even on air cooling, as is indicated in Mr. Emerson's paper. They are, therefore, somewhat sensitive to thermal shock and they require suitable preheat and stress relief or other suitable heat treatment after welding or following other hot fabricating operations where they might be air cooled from their transformation temperatures. In order to make fabricating operations somewhat more flexible, to make them less crack sensitive during welding procedures, and to improve yield and reduce conditioning time during manufacture, it was found expedient, with increasing experience, to use moderate amounts of aluminum during deoxidation, and it has become common practice to use up to 1 lb of aluminum per ton in manufacture. There are exceptions to this for certain piping installations in main steam leads but not insofar as refinery tubes and superheater tubes are concerned. Chromium steels, at this chromium level, tend to become fine grained with No. 3 to 6 ASTM austenitic grain size without aluminum deoxidation while small additions of aluminum produce a fine No. 6 to 8 ASTM austenitic grain size. It is not believed that aluminum in moderate amounts up to 1 lb per ton seriously affects creep or rupture properties of the steel when tested or used in the annealed condition. Our own company's original creep tests were made on the coarse-grained varieties and we later made an extensive series of rupture tests on the $2\frac{1}{4}$ per cent chromium, 1 per cent molybdenum steel representative of a fine-grained heat. This does not answer the question raised by Mr. Emerson as to the effect of the structural grain size on high-temperature creep or rupture properties of these steels, and further work would appear to be necessary to evaluate this effect. In any case, large diameter, heavy wall steam pipe of these alloys is likely to have a considerably coarser structural grain size than smaller oil still or superheater tubes.

Both of these steels are relatively stable structurally as contrasted to those containing lower amounts of chromium and molybdenum at 1000 F, or higher, and they appear, from the data given in Mr. Emerson's aging tests, to be relatively stable in the normalized-and-tempered condition at 1050 F, with indication that they are less so at 1150 F. The precipitation effect described has been noted in these and other molybdenum-containing steel when heated and is observed in annealed steels. It perhaps explains in part their enhanced resistance to plastic flow under stress at elevated temperature. These two steels exhibit a wide range of properties on air cooling and tempering, and Mr. Emerson properly raises the question whether we are taking full advantage of their possible properties by using them in the fully softened condition. We have given some consideration to this question in connection with superheater fabrication and, while difficult, it is not entirely impractical to heat treat completed sections of several loops by normalizing from temperatures in excess of 1700 F followed by tempering at an appropriate temperature. Engineering-wise, we feel that it is better to compromise between having reasonable ductility under low strain rates with moderate rupture strength rather than to do excessive grain coarsening and thereby obtain maximum resistance to flow but destroy the ability of the steel to undergo appreciable deformation without encountering brittle intergranular fracture.

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[7] 1951 Supplement to Book of ASTM Standards, Part 1.

TABLE IV.—PRELIMINARY TESTS ON EFFECT OF HEAT TREATMENT ON STRESS-RUPTURE PROPERTIES.
Composition: C 0.11, Mn 0.50, Si 0.44, Cr 2.24, Mo 0.93.

Specimen	Heat Treatment	Temperature, deg Fahr	Stress, psi	Fracture Time, hr	Elongation in 1 in., per cent	Reduction of area, per cent
No. 43CA	1575 F, 1 hr F.C.	1000	28 000	118	57.0	85.2
No. 43CB	1650 F, 1 hr A.C.	1000	28 000	389	13.7	31.4
No. 43CC	1650 F, 1 hr A.C. 1100 F, 1 hr A.C.	1000	28 000	392	16.5	25.8
No 43CD	1650 F, 1 hr A.C. 1200 F, 1 hr A.C.	1000	28 000	414	12.6	23.5
No. 43CE	1650 F, 1 hr A.C. 1350 F, 1 hr A.C.	1000	28 000	164	27.2	53.7
No. 43CF	2000 F, 1 hr A.C.	1000	28 000	694	2.2	2.7

TABLE V.—STRESS-RUPTURE PROPERTIES OF ANNEALED 2¼ CHROMIUM, 1 MOLYBDENUM STEEL.

Material No. 246 Coupon	Temperature, deg Fahr	Stress, psi	Time for Rupture, hr	Elongation in 2 in. at Rupture, per cent	Reduction of Area, per cent
No. R-6	1000	17 000	9 240	60.1	75.2
No. R-1	1000	20 000	1 699	82.8	83.0
No. R-2	1000	25 000	144	56.2	82.5
No. R-3	1000	30 000	44	53.1	83.5
No. R-4	1000	35 000	29	53.1	81.5
No. R-5	1000	40 000	25	53.9	81.0
No. R-7	1100	8 000	29 286	30.0	86.0
No. R-8	1100	10 000	10 386	50.0	83.7
No. R-9	1100	15 000	495	57.0	87.2
No. R-10	1100	20 000	90	82.8	90.5
No. R-11	1100	25 000	21	65.6	87.2
No. R-17	1200	4 000	15 189	[a]	86.0
No. R-16	1200	5 000	4 490	[a]	...
No. R-15	1200	6 000	3 329	78.1	92.0
No. R-13	1200	8 000	809	32.3	89.9
No. R-12	1200	9 000	369	45.3	...
No. R-14	1200	10 000	212	50.0	91.0
No. R-18	1200	15 000	29	70.0	91.5

[a] Reference marks scaled off.

Analysis: C 0.12, Mn 0.46, Si 0.36, S 0.014, P 0.012, Cr 2.26, Mo 1.04.

Annealed 1650 F 1 hr, furnace cooled 40 F per hr to 1400 F, thereafter cooled in furnace.

McQuaid-Ehn austenitic grain size ASTM No. 7 to 8.

Room temperature mechanical properties before testing: tensile strength 81,200 psi. Yield strength, 0.2 per cent offset, 48 000 psi. Elongation in 2 in. 31.0 per cent. Reduction of area 68.8 per cent. Brinell hardness 161.

While we hope to do some additional testing work along this line we can cite some preliminary data recently secured by the Barberton Works Laboratory, Babcock & Wilcox Co., relating to the effect of heat treatment on fracture time of $2\frac{1}{4}$ chromium, 1 molybdenum steel. The specimens were all taken from the same material and were subjected to the heat treatments indicated in the accompanying Table IV. These data show the annealed alloy (43CA) has a low fracture time but excellent ductility whereas the specimen (43CF) grain coarsened at 2000 F has the maximum time to rupture but its ductility is seriously lowered. This circumstance would likely be worsened at longer times to fracture on testing under lower stress. The normalized-and-tempered specimens show improved rupture strength in terms of time but at sacrifice of ductility. With tempering treatment at 1350 F, the fracture time begins to approach that for the annealed specimen. The ductility at rupture even on quite extended tests, for annealed alloy, may be noted from the data given in Table V. These are from a full series of rupture tests made on this alloy on commercial material by The Babcock & Wilcox Tube Co.

At the temperatures under discussion, that is, 1000 to 1100 F, the engineer appears to have a choice of obtaining a piping material which has been treated for maximum resistance to creep or rupture but has very limited ability to withstand creep strain or he may elect to obtain a material in a condition offering more moderate strength but able to stand considerable deformation before encountering damage. In substance, by improving resistance to deformation within grains, the grain boundaries are forced to suffer the deformation and they have but little ability, at these temperatures, to take such deformation without initiating intergranular fracture. This phenomenon is common to many of the alloy piping materials and is not confined to the two steels under discussion. In superheater and oil still tubes, there is some uncertainty as to exact metal temperature, and being under variable conditions of heat input and cleanliness of surface, it has been felt that annealed steels would offer best over-all service. This is aside from the fabricating problems involved. This situation may not hold for 1000 to 1050 F piping systems where temperature conditions are more precisely known. The individual elements of fabricated pipe may be precisely heat treated before welding into the assembled steam line and thereafter stress-relieved at appropriate temperature which would correspond with the original tempering temperature used. In consequence, Mr. Emerson's suggestions deserve careful consideration and study. The matter is not without precedent since General Electric Co. has prescribed such special treatments for various steels including molybdenum-vanadium and chromium-molybdenum-vanadium piping steels.[8, 9] Similar heat treatments have also been used in foreign power plants. ASA Piping Code design stresses would have to be increased from present values to permit advantage to be taken of an improvement which might be gained by general use of normalizing-and-tempering heat treatments. Much more long-time stress-rupture data will be necessary before taking such a step, and the stability of microstructure and mechanical properties on more extended exposure should be further explored.

As Mr. Emerson suggests, the steels should be heat treated in such manner as to retain a reasonable balance of mechanical properties at ambient temperature. Perhaps necessary studies to answer the questions raised in Mr. Emerson's paper could be sponsored as an industry matter.

MR. J. L. VAN ULLEN (*by letter*).[10]—The relative stability of high-temperature piping steels deserves the careful analysis presented by Mr. Emerson. In the operation of essentially isothermal high-temperature machines, the change

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[8] E. L. Robinson, "Some 1000 F Steam Pipe Materials," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 70, p. 855 (1948).

[9] A. W. Rankin and W. A. Reich, "High Temperature Properties and Characteristics of a Ferritic Steam-Piping Steel," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 73, p. 891 (1951).

[10] Schenectady Works Laboratory, General Electric Co., Schenectady, N. Y.

in physical properties with time-at-temperature can be evaluated more closely than where the temperature fluctuates. Hence it does not always appear economical to utilize a steel in a weak high-temperature condition if the decrease in strength with time of the heat-treated steel is moderate and can be forecast.

The service tempering data for normalized $2\frac{1}{4}$ per cent chromium, 1 per cent molybdenum steel shown in Fig. 8 of the paper are informative and can possibly be extended to predict the hardness after 100,000 hr services at 1050 F and 1150 F as shown in the accompanying Fig. 15.

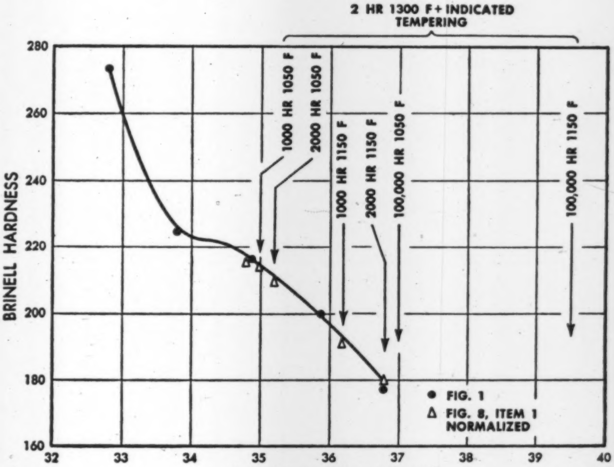

FIG. 15.

The tempering data of Fig. 3 for normalized $2\frac{1}{4}$ per cent chromium, 1 per cent molybdenum steel were used to construct the master tempering curve, Fig. 15, of parameter M as Brinell hardness. The parameter concept[11] assumes that time and temperature are codependent during tempering and related by the equation:

$$M = T(C + \log t) \ldots\ldots (1)$$

where T is the absolute temperature in degrees Rankine, C is a constant taken as 19.5, and t is the time expressed in hours.

Since a portion of Fig. 8, Item No. 1, represents the material initially normalized and tempered 2 hr at 1300 F plus various increments of tempering at 1050 F and 1150 F, the total tempering after 2000 hr at 1050 F is the sum of the 1300 F and 1050 F treatment. When tempering is composed of separate stages, each at a fixed temperature, the total tempering at the end of each stage can be represented by:

$$M = T \log(\Delta t\, 10^{c} + 10^{M_0/T}) \ldots (2)$$

where Δt is the time spent at temperature T and M_0 is the value of the parameter at the start of this stage.

If the tempering parameter is valid, then the data shown in Fig. 3 can be calculated from Eq 2. As an example, 2 hr at 1300 F + 1000 hr at 1150 F, represents a parameter value of 35,700.

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[11] J. H. Hollomon and L. D. Jaffe, "Time-Temperature Relationships in Tempering Steels," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 162, pp. 223–249 (1945).

The master curve, Fig. 15 shows the hardness should be 192 Brinell, which agrees with the experimentally determined value of about 192 in Fig. 8. Other check points agree with the experimental data. The calculated hardness after 100,000 hr at 1050 F is 175 Brinell, but insufficient data are available to predict the hardness after 100,000 hr at 1150. If the author were to temper the normalized 2¼ per cent chromium, 1 per cent molybdenum steel, Item 1, 400 hr at 1350 F or 100 hr at 1400 F, a degree of tempering equivalent to 100,000 hr at 1150 F would be approximated. However, the justification for not relying on strength acquired by normalizing this composition is well supported by the general trend of the data.

Referring to the author's statement under the heading "Background in Use of Steam Piping Materials" that "... in fact, creep or any changes in creep rate are usually the result rather than the cause of changes in metallurgical structure", I do not believe that creep is usually the result rather than the cause of metallurgical changes. Creep or creep rate may be altered by metallurgical changes but creep certainly occurs whether or not there are metallurgical changes of the character typified by graphitization and spheroidization.

The author is to be congratulated on presenting such an interesting and stimulating summary of information on the 2¼ per cent chromium, 1 per cent molybdenum alloy, which is becoming increasingly important in the construction of high-temperature power generating equipment.

MR. EMERSON (*author's closure*).—The author wishes to acknowledge the excellent discussions submitted and considers them collectively as a valuable contribution to this paper.

The discussions of Messrs. Kanter, Newell, and Wilder with respect to the effects of grain size, heat treatment, etc., on the high-temperature rupture strength of the intermediate chromium-molybdenum alloy steels is believed to be most pertinent and do, in general, confirm the author's belief.

In some instances, the extreme scatter of results of high-temperature properties of the low- and intermediate-alloy steels, as found in the literature, are believed to be due principally to an attempt to evaluate such properties on the basis of chemical analysis. Such important factors as McQuaid-Ehn grain size, structural or ferritic grain size, type of heat treatment, etc, which are usually more important than the actual chemical analysis of the material itself, are believed to have been, in these instances, completely overlooked.

The values of 5000 *versus* 9000 psi creep stress for two materials of nominally the same analysis, but varying in deoxidation practice and ferritic grain size, as shown by Mr. Kanter; and the extreme difference in fracture time of 118 *versus* 694 hr obtained from a single heat of 2¼ per cent chromium, 1 per cent molybdenum steel, the specimens differing only in the type of heat treatment, as reported by Mr. Newell, clearly indicates that deoxidation practice, ferritic grain size and heat treatment are most important considerations in the evaluation of high-temperature properties of low- and intermediate-chromium-molybdenum steel.

Mr. Van Ullen's discussion of the "parameter concept" and the ability to predict hardness of a material after 100,000 hr at temperature by this method of calculation is most intriguing and merits considerable thought.

MICROSTRUCTURAL INSTABILITY OF STEELS FOR ELEVATED TEMPERATURE SERVICE*

By G. V. Smith, W. B. Seens, H. S. Link, and P. R. Malenock[1]

Synopsis

This paper describes the effect of exposure for 10,000 hr (about 14 months) at 900, 1050, or 1200 F on microstructure, hardness at room temperature, and notch-impact strength at different temperatures of 18 ferritic or austenitic steels which may be applied in service at elevated temperatures. In the low-alloy ferritic steels carbide spheroidized and a fine precipitate developed. No graphitization was noted. Alloys containing 5 Cr, $\frac{1}{2}$ Mo (Ti) or 9 Cr, 1 Mo showed no significant changes in microstructure. Carbide spheroidized in the 12 Cr, and 17 Cr grades and in the latter sigma also precipitated. In the austenitic steels, both carbide and sigma precipitated in the 18 Cr, 8 Ni (Mo, Cb), 18 Cr, 8 Ni (Ti), and 18 Cr 8 Ni (Cb) grades and carbide in the remaining grades including the 0.03 (max) carbon grades. These changes resulted in impairment of notch impact strength in about two-thirds of the exposures and improvement in about 10 per cent, whereas the remaining 25 per cent remained essentially unaffected in this respect.

During prolonged exposure at relatively high temperatures, most metals and alloys undergo changes in microstructure. In steels, the kinds of change are quite varied and, depending on the particular steel and its prior history, may include one or more of the following: carbide spheroidization, graphitization, recrystallization, grain growth, and transformation or precipitation reactions of one sort or another, such as the development of the sigma phase in the high-chromium or chromium-nickel steels. In addition to these, certain other changes may occur, such as strain aging, temper embrittlement, so-called 885 F embrittlement in chromium steels, and recovery, which are not readily, if at all, detected in the microstructure.

These structural changes affect the material properties, not only at the temperature of exposure, but at other temperatures as well. The changes are of special interest in the case of metals used in service at elevated temperature, for when they occur the properties change continuously throughout the service life; the metal removed from service differs from that which was put in service, thus complicating the problem of design. This variation of material properties with time is one of the principal reasons why long-time behavior cannot be predicted from tests of short duration. Accordingly, an important aspect of the evaluation of metals for elevated temperature service is the study of the influence of time and temperature on structure.

The present paper presents the results of a study of the changes in microstructure of 18 steels (Table I) suitable for elevated temperature service resulting from exposure for 10,000 hr at 900, 1050, or 1200 F, and of the effects of these changes upon hardness and upon the variation of the notch-impact strength with temperature. These data supplement other results currently being obtained by the National Tube Co. with similar, and in some cases identical, steels on the effect of such exposure on the microstructure and creep-rupture strength. Some of the initial results of this latter study have already been reported (**1**, **2**).[2]

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* Presented at the Fifty-fourth Annual Meeting of the Society, June 18–22, 1951.

[1] Research Laboratory, United States Steel Company, Kearny, N. J.

MATERIALS AND PROCEDURE

Eighteen steels of interest for use in elevated temperature service, having the chemical compositions listed in Table I, were chosen for test. These were selected to provide a gradation of alloy content from a very low level, ½ Mo, up to, and including, the various 18 Cr, 8 Ni type alloys. Except for the ½ Mo alloy, only one heat of each type is represented, and although the heats are believed to be representative of the grades in question, the results should be interpreted with the caution that they may not necessarily be

TABLE I.—CHEMICAL COMPOSITION OF STEELS, PER CENT.

Type of Steel	C	Mn	P	S	Si	Ni	Cr	Mo	N	Al	Other
½ Mo (Si deoxidized)	0.16	0.85	0.020	0.019	0.24	...	...	0.51	0.005	0.010	
½ Mo (Si and Al deoxidized)	0.13	0.52	0.012	0.021	0.16	...	...	0.52	0.005	0.044	
½ Cr, ½ Mo	0.15	0.50	0.010	0.022	0.21	0.07	0.48	0.53	0.005	0.008	
1 Cr, ½ Mo	0.12	0.45	0.013	0.015	0.74	...	1.13	0.50	0.011	0.010	
3 Cr, 1 Mo	0.11	0.45	0.011	0.021	0.39	0.21	3.21	0.97	0.016	0.001	
5 Cr, ½ Mo	0.13	0.42	0.030	0.017	0.22	...	4.69	0.47	0.021	0.004	
5 Cr, ½ Mo (Ti)	0.05	0.40	0.006	0.011	0.31	...	5.19	0.51	0.007	0.075	0.55 Ti
9 Cr, 1 Mo	0.09	0.44	0.022	0.021	0.65	0.28	8.94	0.89	0.025	0.017	
12 Cr (type 410)	0.06	0.45	0.008	0.017	0.32	0.07	12.24	0.02	0.029	0.009	
12 Cr, ½ Mo	0.07	0.40	0.027	0.007	0.29	0.27	12.26	0.49	0.029	0.004	
17 Cr (type 430)	0.10	0.41	0.021	0.014	0.46	0.36	17.00	...	0.076	0.006	0.13 Cu
18 Cr, 8 Ni (type 304)	0.05	0.53	0.008	0.005	0.61	10.72	18.53	...	0.028	0.005	
18 Cr, 8 Ni (0.03 C)	0.028	0.61	0.011	0.009	0.55	10.35	19.30	...	0.032	0.004	
18 Cr, 8 Ni (Mo) (type 316)	0.07	1.53	0.036	0.007	0.51	12.80	16.76	2.15	0.053	0.007	0.05 Cu
18 Cr, 8 Ni (Mo) (0.03 C)	0.022	1.47	0.013	0.023	0.34	10.68	17.56	2.00	0.032	0.005	
18 Cr, 8 Ni (Mo, Cb)	0.05	0.79	0.018	0.006	0.37	14.16	18.68	2.15	0.063	0.016	0.51 Cb
18 Cr, 8 Ni (Ti) (type 321)	0.07	0.52	0.021	0.003	0.39	10.40	17.97	...	0.011	0.069	0.58 Ti
18 Cr 8 Ni (Cb) (type 347)	0.06	1.43	0.018	0.006	0.44	11.16	17.80	...	0.027	0.010	0.77 Cb

TABLE II.—INITIAL HEAT TREATMENT AND RESULTING GRAIN SIZE OF STEELS.

Type of Steel	Heat Treatment	Grain Size[a] (ASTM Scale)
½ Mo (Si deoxidized)	1650 F, air-cooled	5 to 8
½ Mo (Si and Al deoxidized)	1650 F, air-cooled	*8;* 3 to 4 (few)
½ Cr, ½ Mo	1650 F, air-cooled	*8;* 4 to 5
1 Cr, ½ Mo	1650 F, air-cooled	7 to 8
3 Cr, 1 Mo	1550 F, cooled at 50 F per hr	7 to 8
5 Cr, ½ Mo	1550 F, cooled at 50 F per hr	7 to 8
5 Cr, ½ Mo (Ti)	1375 F, cooled at 50 F per hr	7 to 9; band of 3 to 5 in some sections
9 Cr, 1 Mo	1550 F, cooled at 50 F per hr	7 to 9
12 Cr (type 410)	1525 F, slow-cooled to 1100 F, then air-cooled	7 to 8; 4 to 5
12 Cr, ½ Mo	1450 F, air-cooled	7 to 9
17 Cr (type 430)	1425 F, air-cooled	7 to 9
18 Cr, 8 Ni (type 304)	1900 F, water-quenched	4 to 7
18 Cr, 8 Ni (0.03 C)	1900 F, water-quenched	5 to 6
18 Cr, 8 Ni (Mo) (type 316)	1950 F, water-quenched	3 to 5
18 Cr, 8 Ni (Mo) (0.03 C)	1950 F, water-quenched	4 to 6
18 Cr, 8 Ni (Mo, Cb)	1950 F, water-quenched	8
18 Cr, 8 Ni (Ti) (type 321)	1900 F, water-quenched; 1550 F for 2 hr, air-cooled	8
18 Cr, 8 Ni (Cb) (type 347)	1900 F, water-quenched; 1550 F for 2 hr, air-cooled	8 to 9

[a] Ferritic or austenitic depending on predominant structure at room temperature. Grain sizes in accordance with the following ASTM methods:
Standard Classification of Austenite Grain Size in Steels (E 19 – 46), 1949 Book of ASTM Standards, Part 1, p. 1271.
Tentative Methods for Estimating the Average Ferrite Grain Size of Low-Carbon Steels (E 89 – 50 T), 1950 Supplement to Book of ASTM Standards, Part 1, p. 304.

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[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 912.

representative of other heats; it should also be borne in mind that variation is to be expected in any one grade. One of the factors causing such variation is deoxidation practice, and two ½ Mo steels, one deoxidized with silicon alone, the other with both silicon and aluminum were included to reveal the nature of this effect. The deoxidation practice of all except a few of the remaining steels is not known with certainty, but may generally be inferred from the chemical composition. Thus, the ½ Cr, ½ Mo steel was apparently deoxidized with silicon with little, if any, aluminum added.

The steels were made available either as heat-treated ½-in. thick plate (18 Cr, 8 Ni (Mo) and 12 Cr steels) which were subsequently sawed to ½-in. widths or as 1-in. square or round bars (all other steels). To conserve material, the 1-in. bars were hot-forged to ½-in. square section and reheat treated. The heat treatments employed and the resulting grain sizes are given in Table II; both are representative of commercial practice.

The steels were exposed for 10,000 hr at 900, 1050, or 1200 F in three box-type electric furnaces described by Wilder and Tyson (1). Temperature uniformity was within ±7 F. The bars were air-cooled at the completion of the exposures. The specimens were exposed, without stress, in the heat-treated (normalized or annealed) condition, as recorded in Table II. Although in theory, either stress or plastic deformation should accelerate microstructural changes, in practice, the effects of these variables when of the magnitude of interest in creep service seem to be minor or undetectable (3).

Standard keyhole-notch Charpy impact test specimens were machined from the ½-in. square bars after exposure. The specimens were tested in a Riehle machine having 2-mm radius on striker and anvil edges (European standard), thus differing from ASTM Tentative Methods: E 23 - 47 T.[3] It has been demonstrated that ASTM specifications result in abnormally high energy values at high energy levels (tough specimens), so the results of these tests should be judged accordingly.

Since in standard notch-impact tests, such as are reported here, specimen geometry and rate of stressing are arbitrary, the numerical values obtained cannot be directly employed in design. Rather, they provide a measure of relative toughness, or of ability to resist fracture, under multi-axial stress and moderately rapid stressing. Further, since many ferritic steels suffer a relatively abrupt decrease in notch-impact strength with decreasing temperature, that is, they undergo a transition from ductile to brittle behavior, the toughness of such steels, at least, cannot be properly evaluated unless the effect of temperature is explored. This transition often occurs near, or below, room temperature.

In the present measurements, the effect of test temperature was explored in the range −315 F to 500 F. Although the austenitic steels do not undergo an abrupt transition, they may in some cases lose toughness with decreasing temperature; therefore, they were likewise tested at various temperatures. In the case of the ferritic steels, an effort was made to encompass the transition range of temperature. Although it may have been desirable in some instances to continue tests to higher temperatures than were employed, this was not done, partly because of lack of material and of the experimental difficulties and also because experience has indicated that notch-impact toughness increases with increasing temperature, at least within the limits of the range of commercial interest. Exception must be made for those plain carbon and low-alloy steels which are

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[3] Tentative Methods of Impact Testing of Metallic Materials (E 23 - 47 T), 1949 Book of ASTM Standards, Part 1, p. 1287; Part 2, p. 1035.

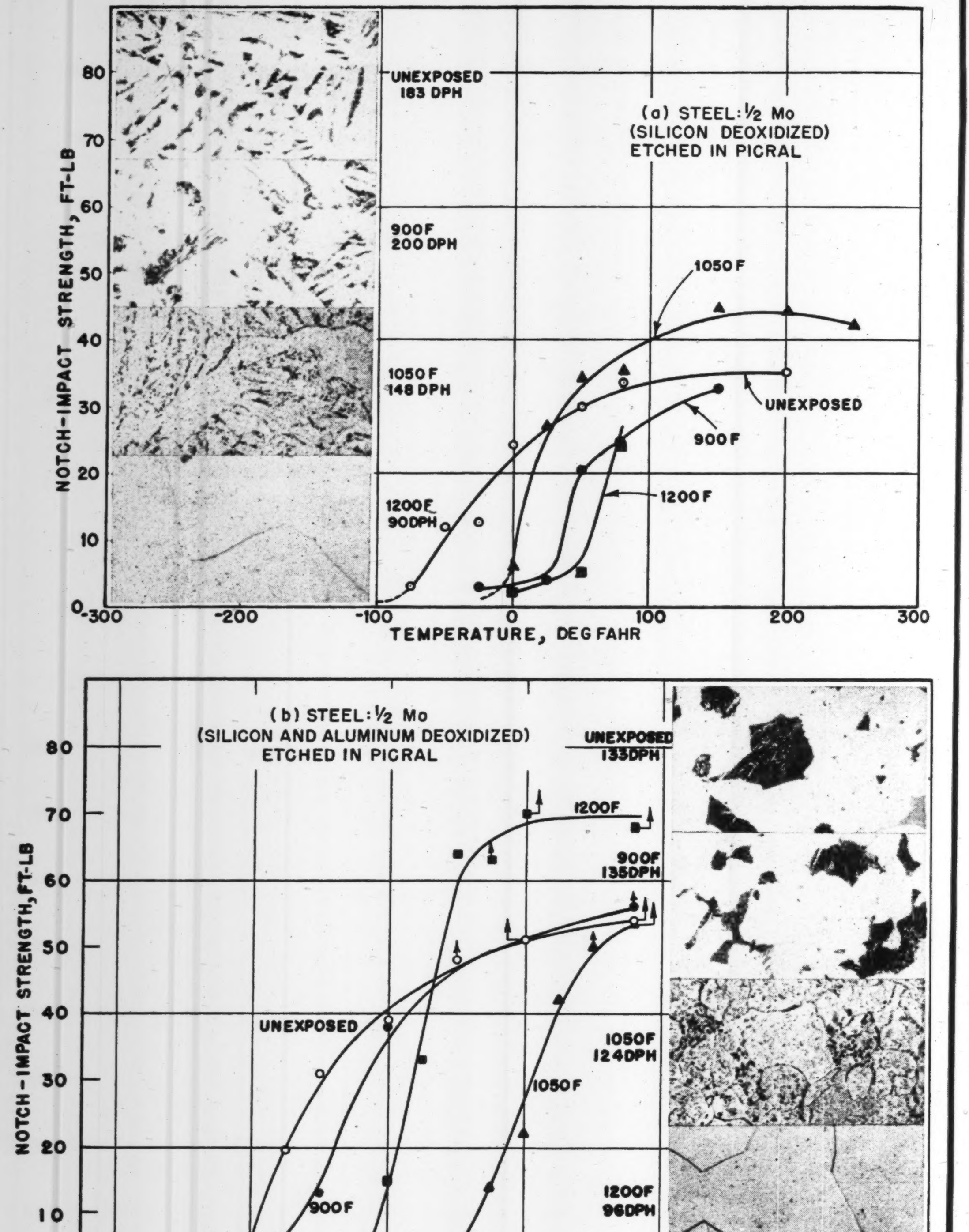

FIG. 1—Effect of 10,000 hr Exposure at 900, 1050, or 1200 F on Microstructure, Hardness and

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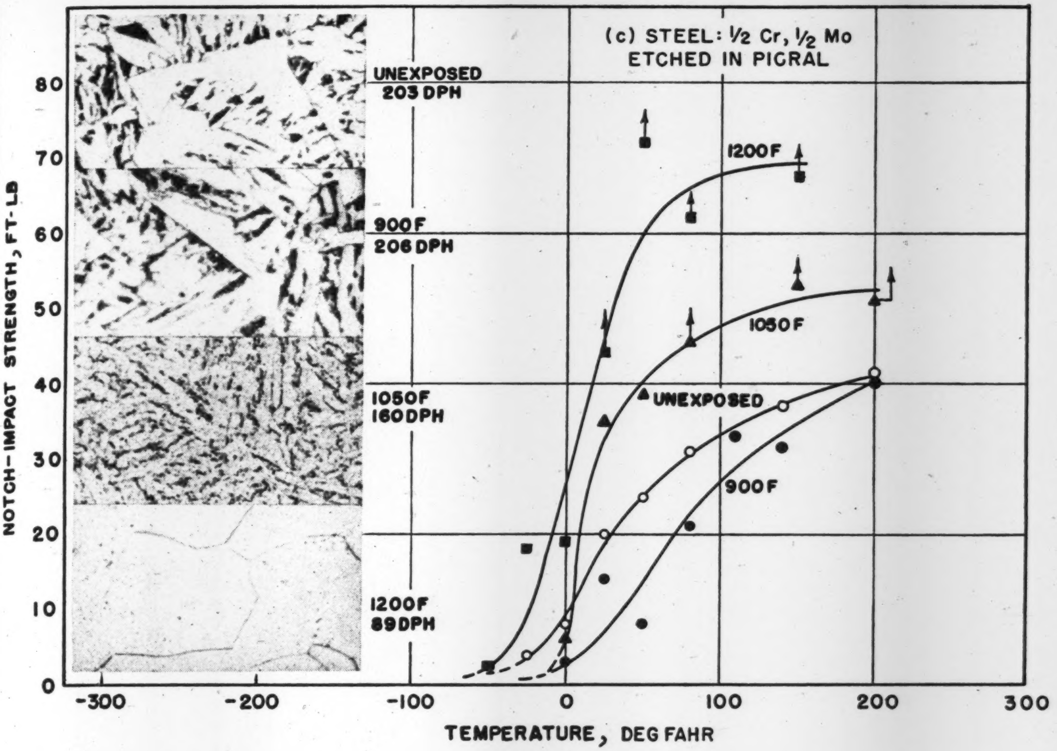

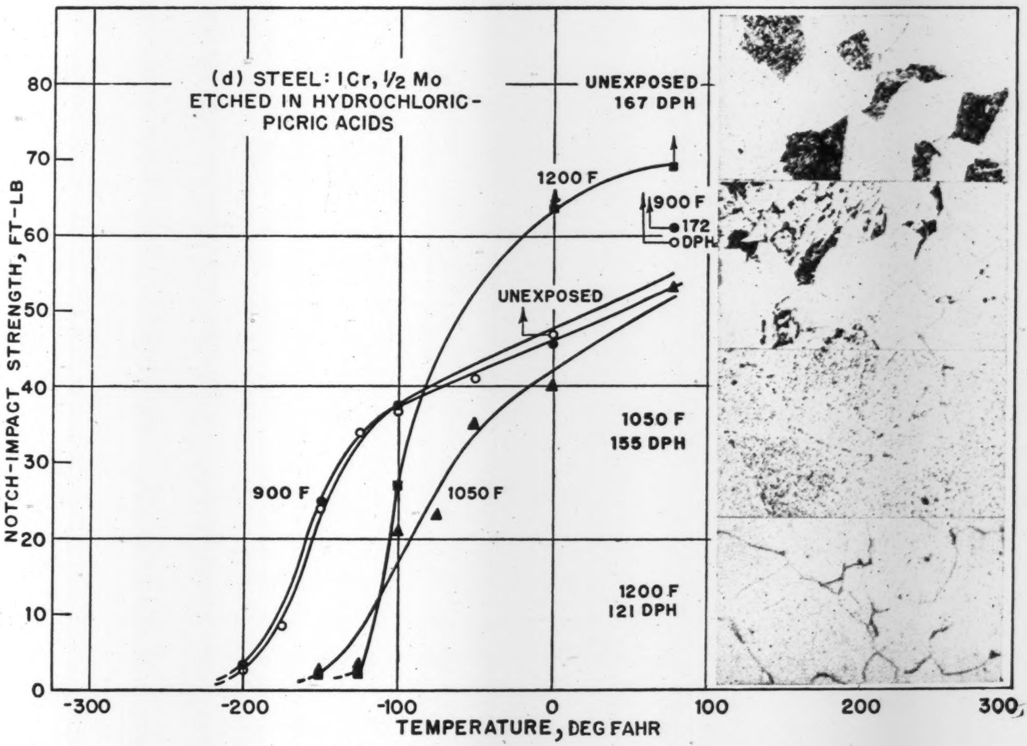

ss and Notch-Impact Strength. Photomicrographs × 1000 (reduced one-half in reproduction).
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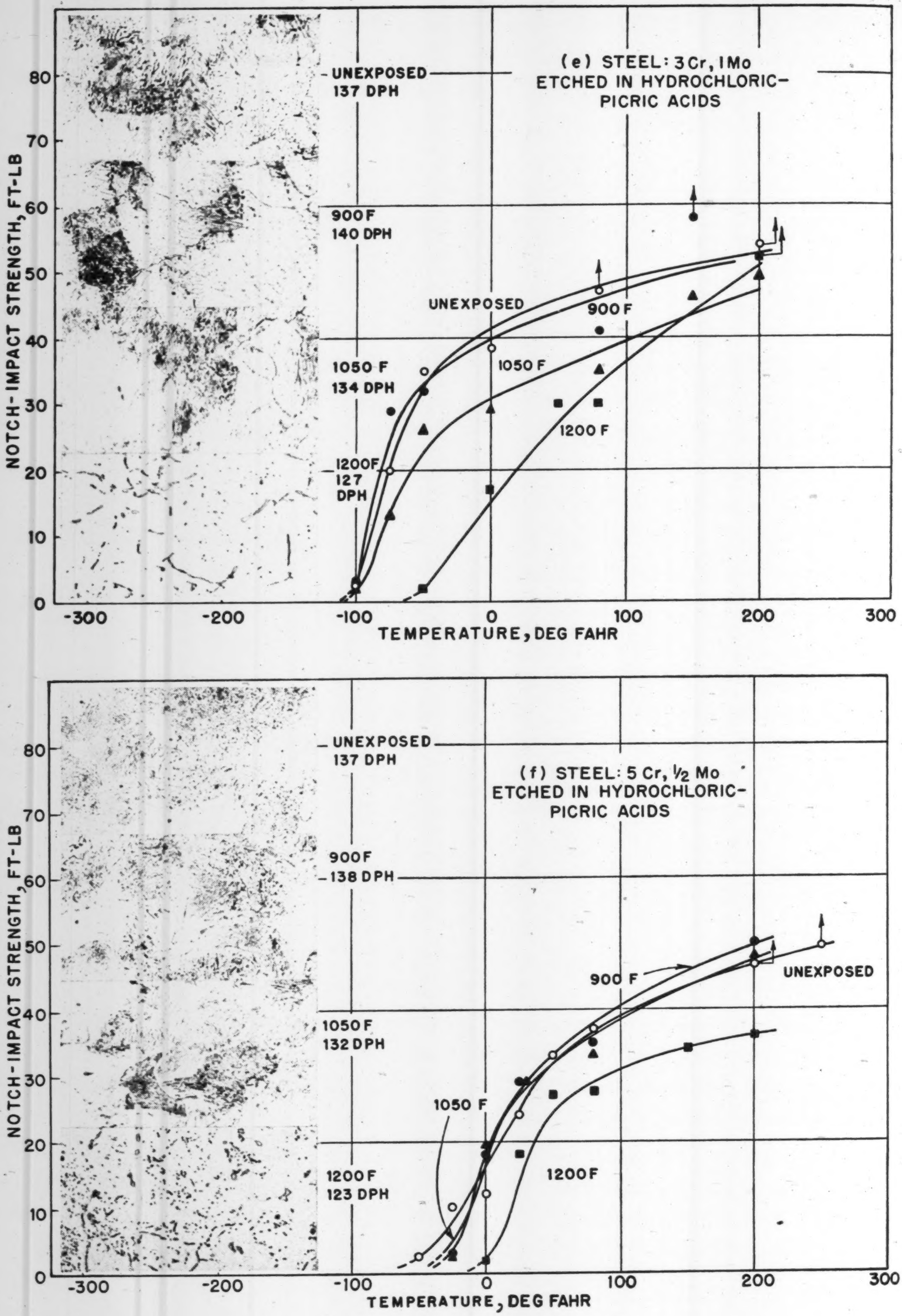

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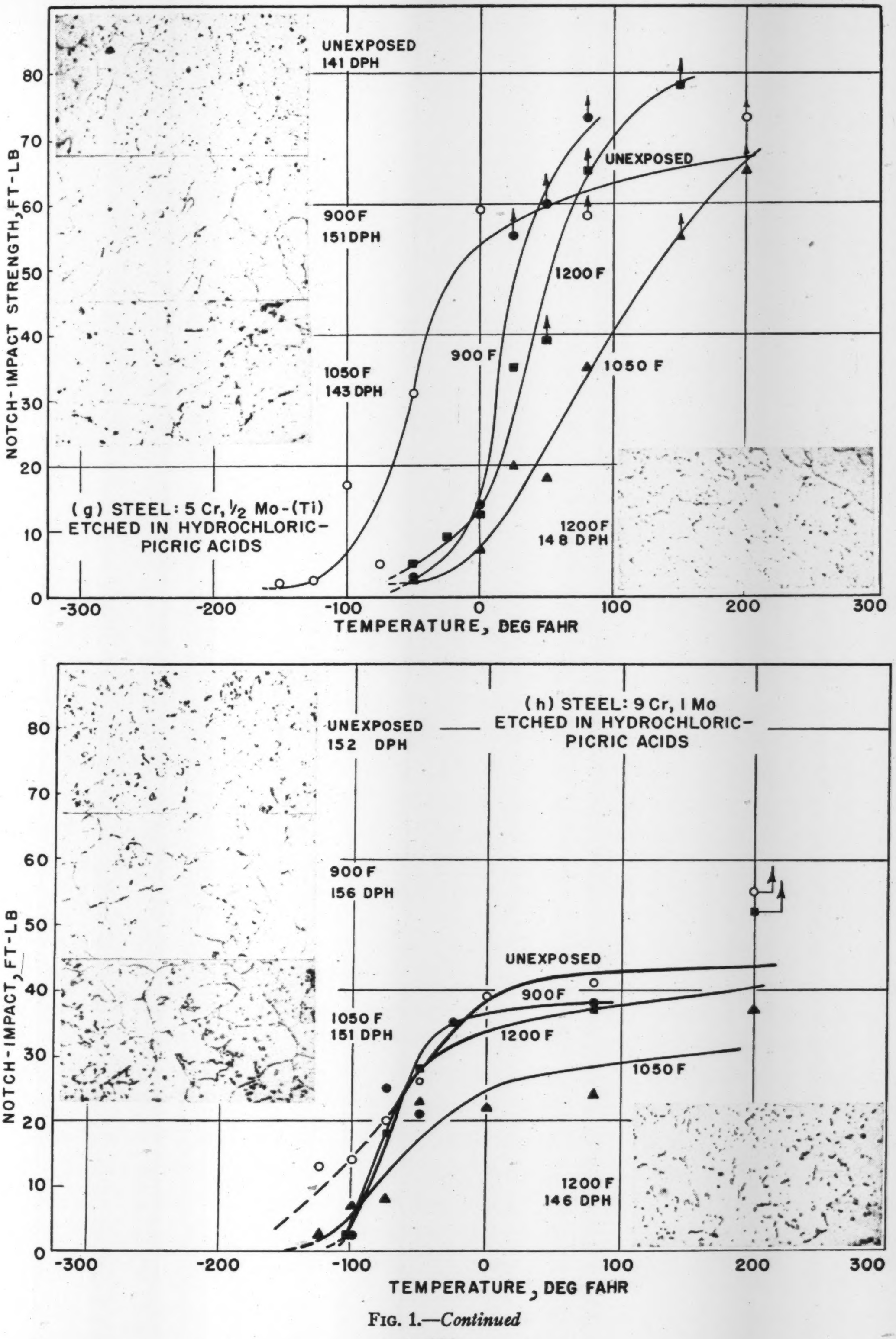

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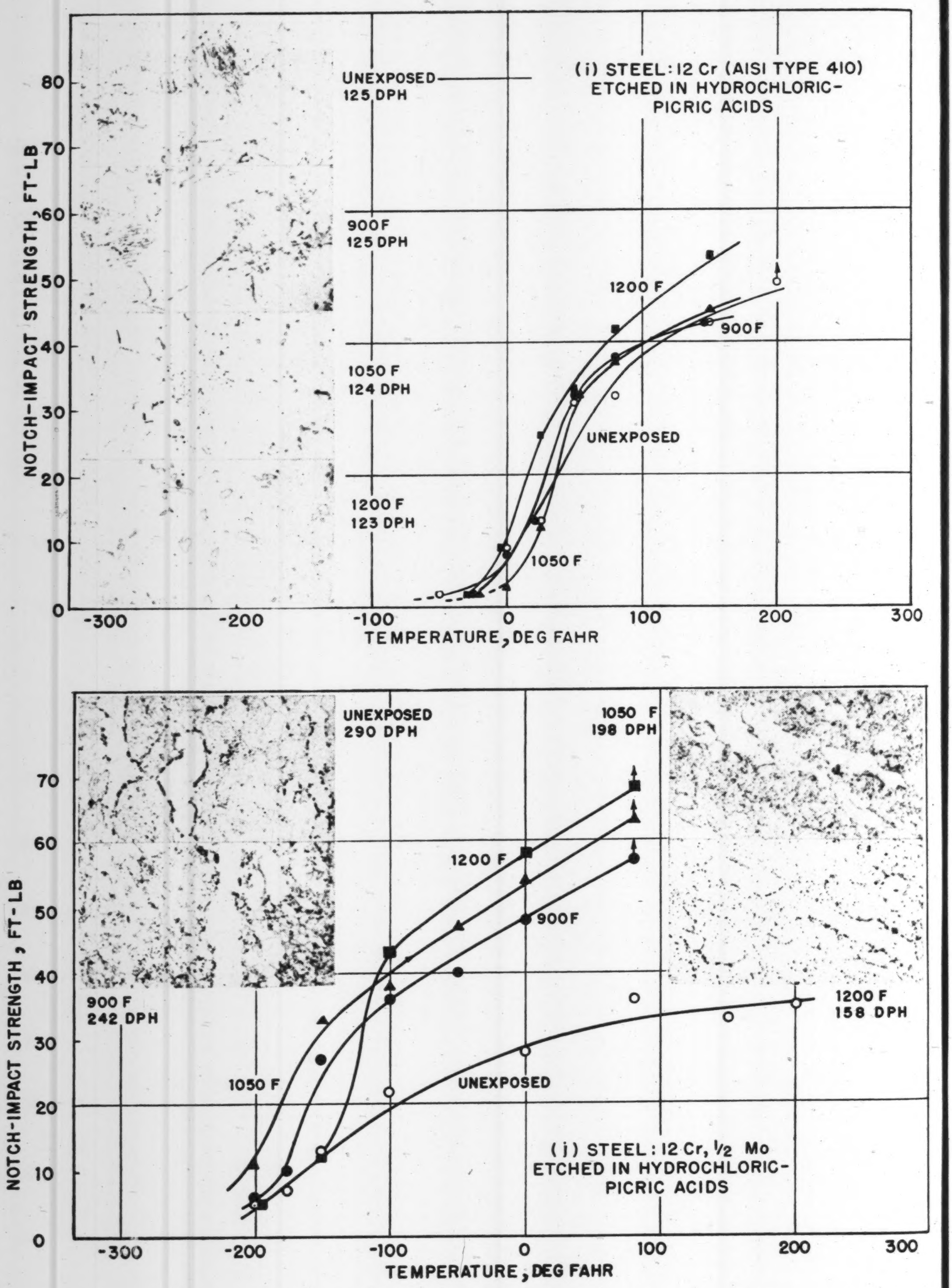

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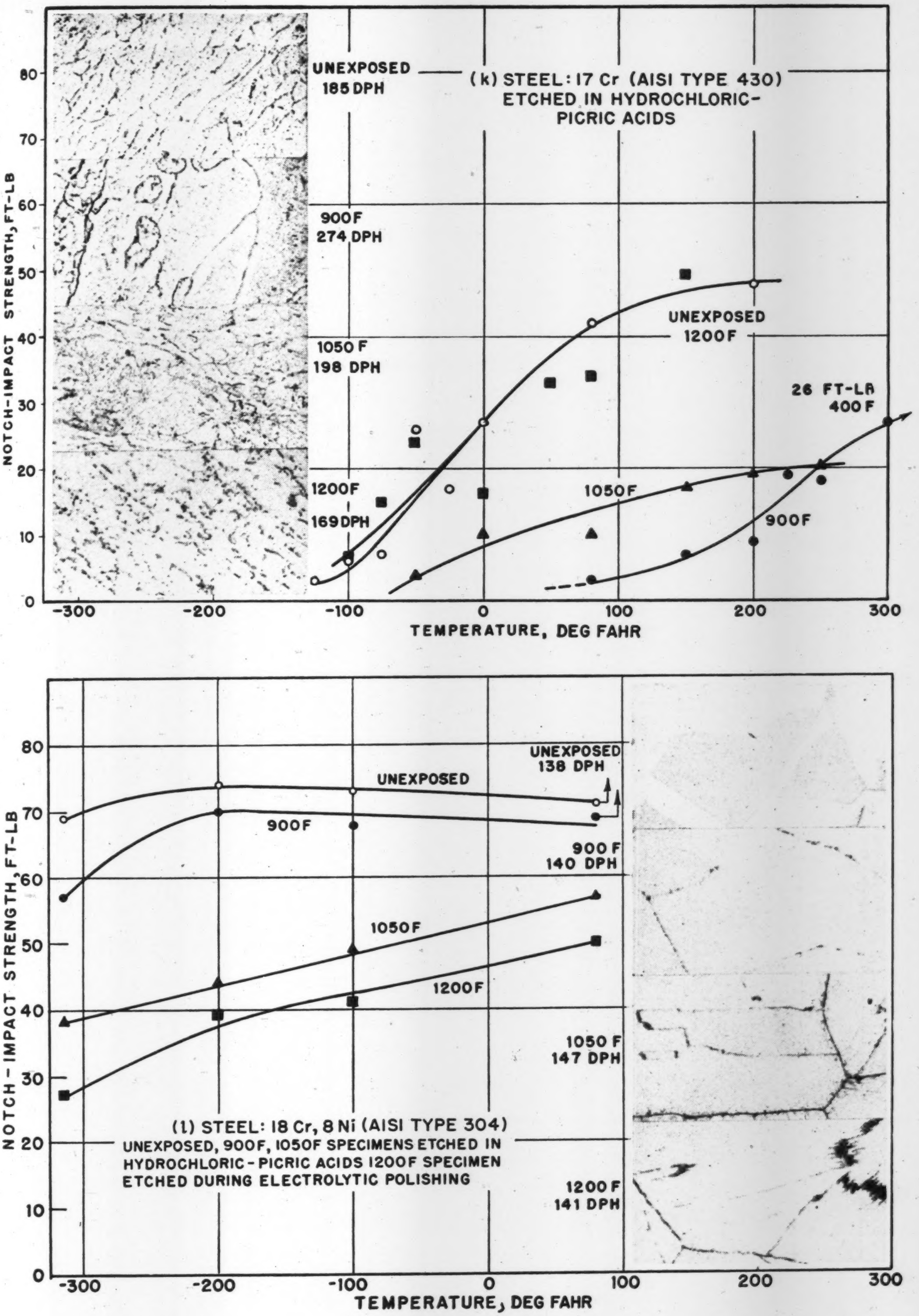


FIG. 1.—*Continued*

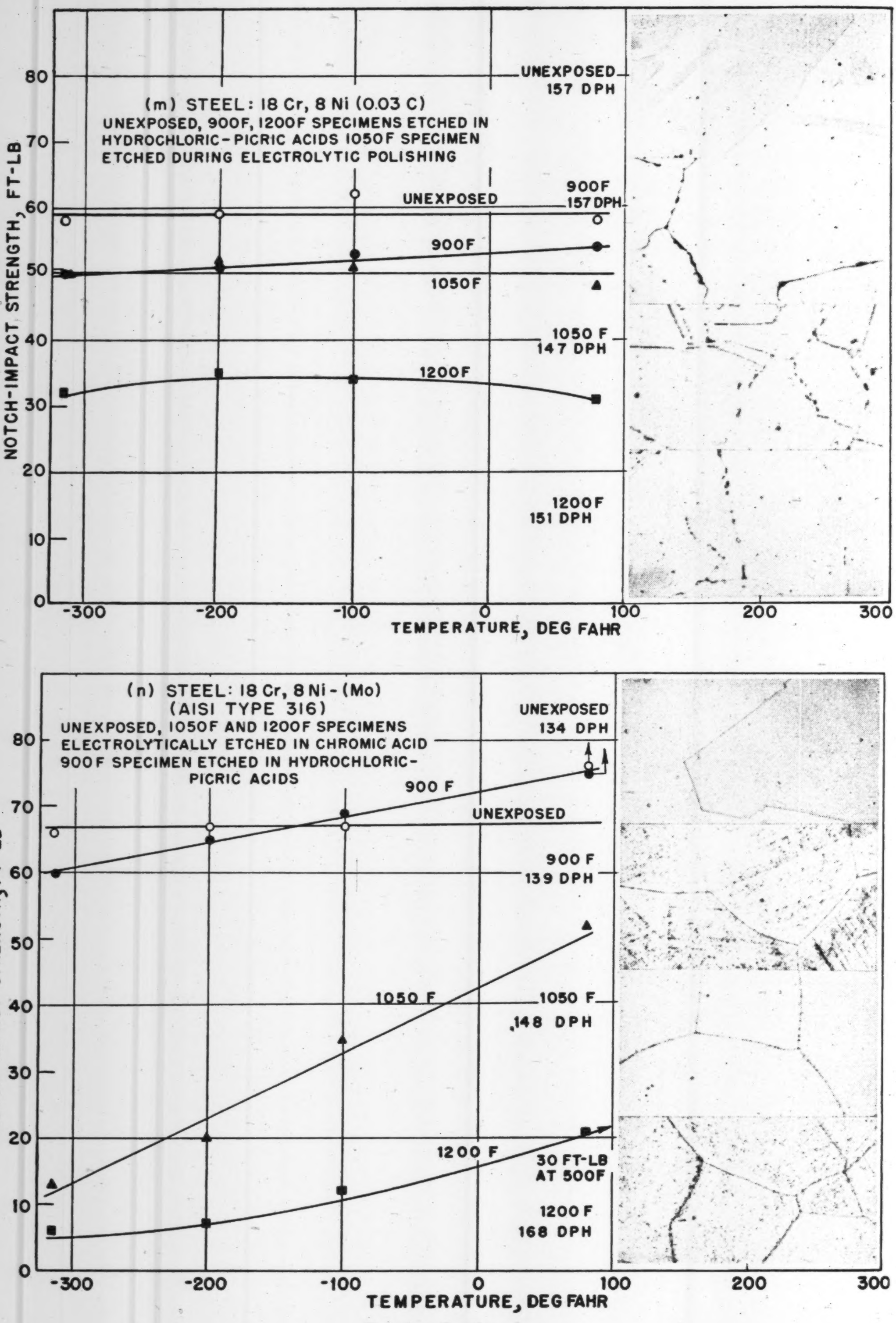


FIG. 1.—*Continued*

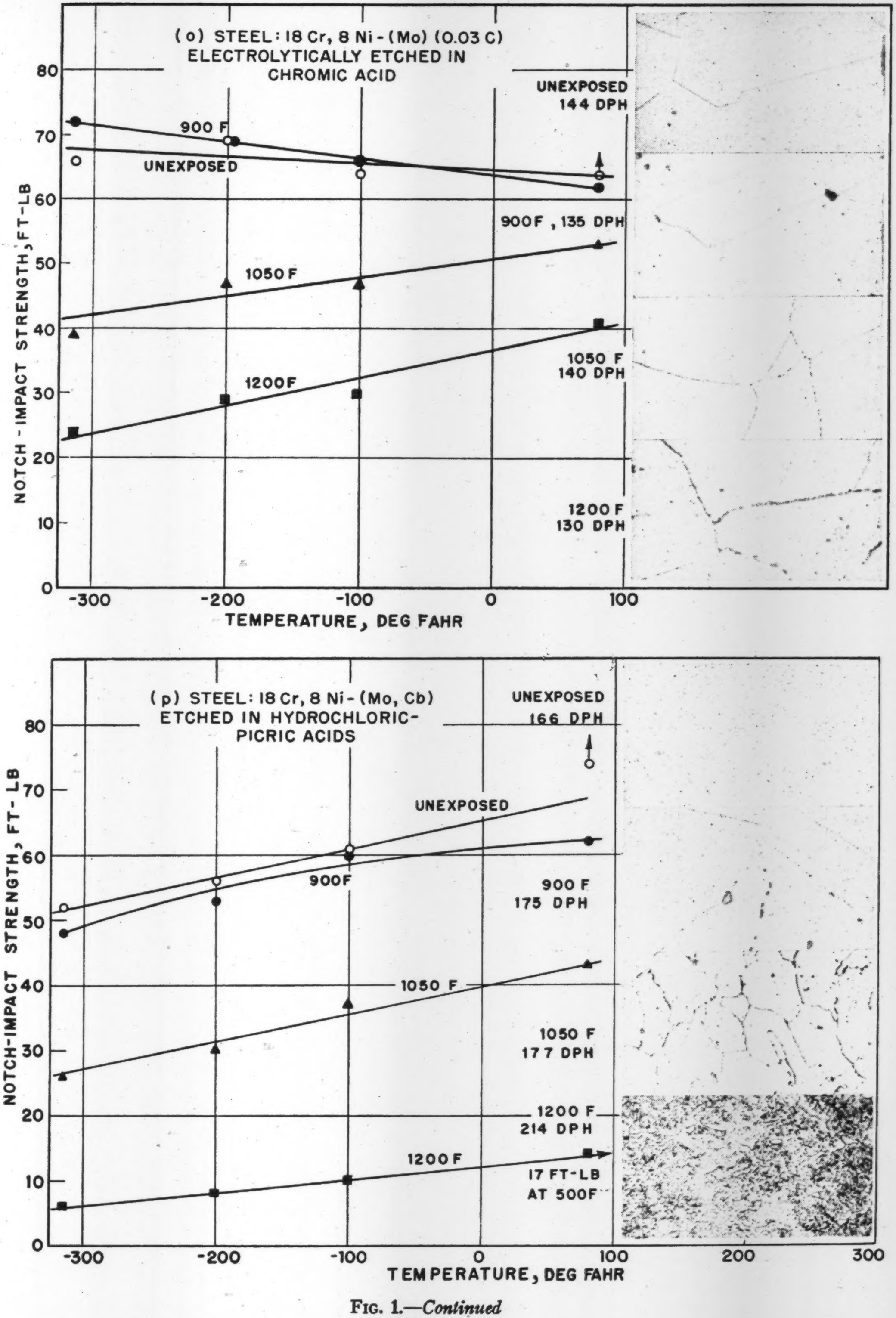
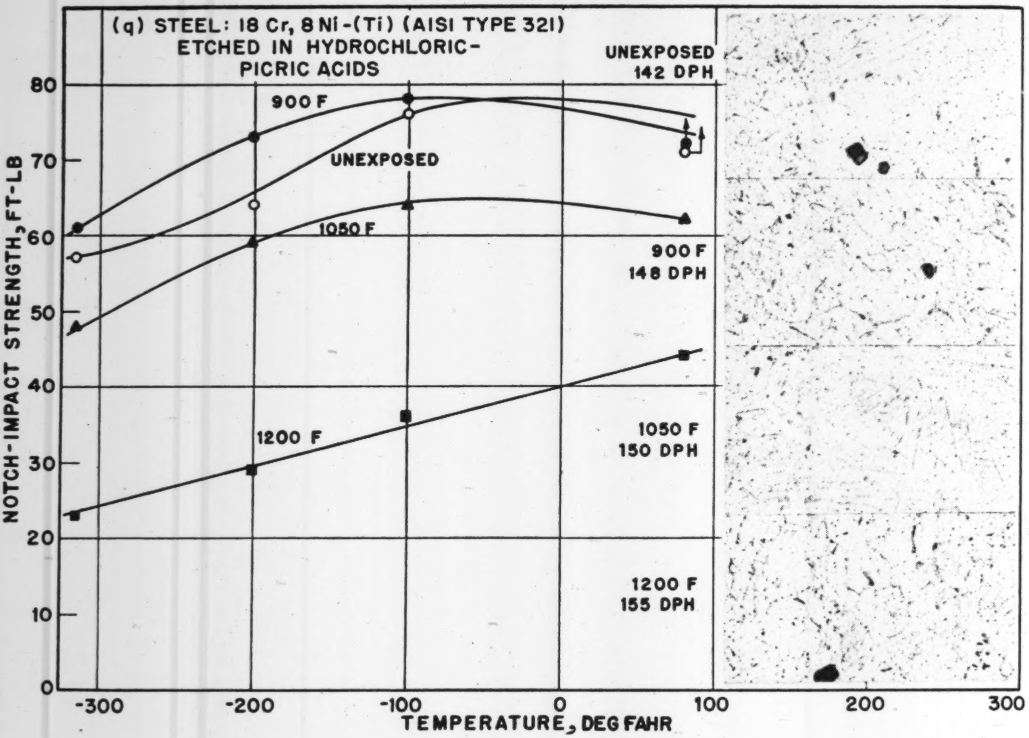


Fig. 1.—*Continued*



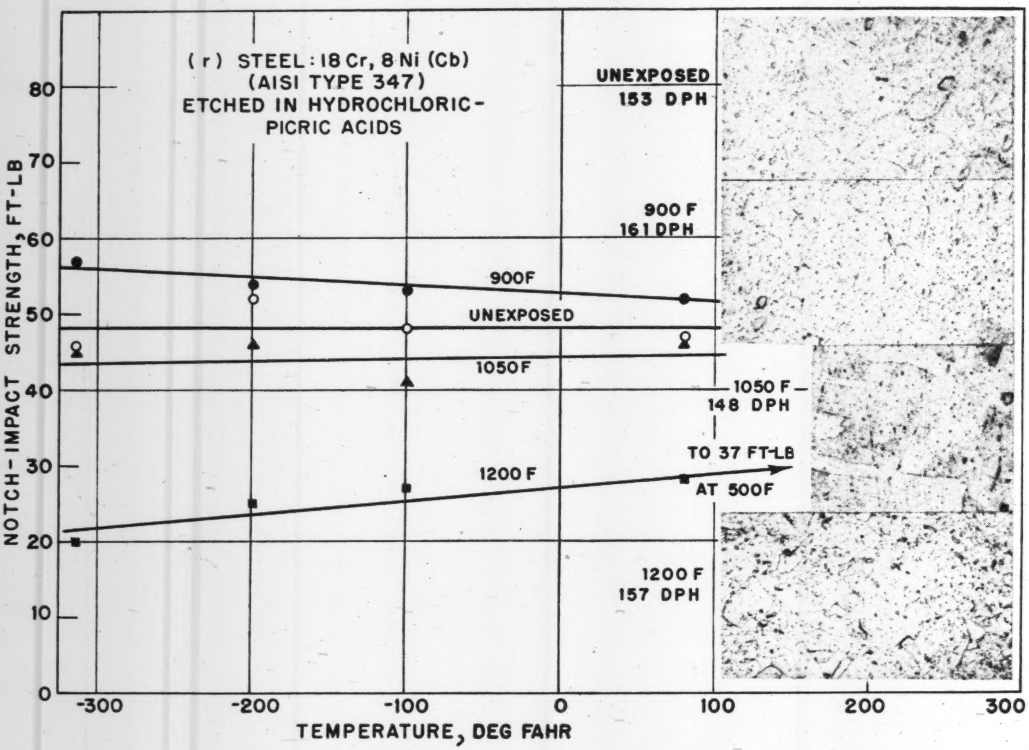


FIG. 1.—*Continued*

TABLE III.—EFFECT OF 10,000 HOURS EXPOSURE ON NOTCH-IMPACT STRENGTH.[a]

Type of Steel	Exposure Temperature		
	900 F	1050 F	1200 F
½ Mo (Si deoxidized)	impaired (2)	impaired (1)	impaired (3)
½ Mo (Si and Al deoxidized)	impaired (1)	impaired (3)	impaired (2)
½ Cr, ½ Mo	impaired	improved (1)	improved (2)
1 Cr, ½ Mo	unaffected	impaired (2)	impaired (1)
3 Cr, 1 Mo	unaffected	impaired (1)	impaired (2)
5 Cr, ½ Mo	unaffected	unaffected	impaired
5 Cr, ½ Mo (Ti)	impaired (1)	impaired (3)	impaired (2)
9 Cr, 1 Mo	unaffected	unaffected	unaffected
12 Cr (type 410)	unaffected	unaffected	unaffected
12 Cr, ½ Mo	improved (2)	improved (3)	improved (1)
17 Cr (type 430)	impaired (2)	impaired (1)	unaffected
18 Cr, 8 Ni (type 304)	impaired (1)	impaired (2)	impaired (3)
18 Cr, 8 Ni (0.03 C)	impaired (1)	impaired (2)	impaired (3)
18 Cr, 8 Ni (Mo) (type 316)	unaffected	impaired (1)	impaired (2)
18 Cr, 8 Ni (Mo) (0.03 C)	unaffected	impaired (1)	impaired (2)
18 Cr, 8 Ni (Mo, Cb)	unaffected	impaired (1)	impaired (2)
18 Cr, 8 Ni (Ti) (type 321)	impaired (1)	impaired (2)	impaired (3)
18 Cr, 8 Ni (Cb) (type 347)	improved	impaired (1)	impaired (2)

[a] Order of impairment or improvement: (1) least changed; (3) most changed. These should not imply degree of change; for this, see Fig. 1 (*a*) to (*r*).

TABLE IV.—EFFECT OF 10,000 HOURS EXPOSURE ON MICROSTRUCTURE.
(Photomicrographs shown in Fig. 1 *a* to *r*.)

Type of Steel	Exposure Trempeature		
	900 F	1050 F	1200 F
½ Mo (Si deoxidized)	slight spheroidization	spheroidization, precipitation	decarburized
½ Mo (Si and Al deoxidized)	slight spheroidization	spheroidization, precipitation	decarburized
½ Cr, ½ Mo	slight spheroidization	spheroidization, precipitation	decarburized
1 Cr, ½ Mo	slight spheroidization	spheroidization, precipitation	spheroidization, precipitation, decarburization
3 Cr, 1 Mo	none	spheroidization	spheroidization
5 Cr, ½ Mo	none	spheroidization	spheroidization
5 Cr, ½ Mo (Ti)	none	none	none
9 Cr, 1 Mo	none	none	none
12 Cr (type 410)	slight spheroidization	spheroidization	spheroidization
12 Cr, ½ Mo	none	spheroidization	spheroidization
17 Cr (type 430)	none	spheroidization, sigma	spheroidization, sigma
18 Cr, 8 Ni (type 304)	carbide	carbide	carbide
18 Cr, 8 Ni (0.03 C)	carbide	carbide,[a]	carbide,[a]
18 Cr, 8 Ni (Mo) (type 316)	carbide	carbide	carbide
18 Cr, 8 Ni (Mo) (0.03 C)	none	carbide	carbide
18 Cr, 8 Ni (Mo, Cb)	carbide	carbide	carbide, sigma
18 Cr, 8 Ni (Ti) (type 321)	none	carbide	carbide, sigma
18 Cr, 8 Ni (Cb) (type 347)	none	carbide, sigma	carbide, sigma

[a] A small amount of a massive phase having the etching characteristics of sigma (though this phase could not be detected by X-ray diffraction) was observed in a central longitudinal segregation, which region showed, in the unexposed condition, a similar segregation of delta ferrite. The photomicrographs of Fig. 1(*m*) were taken in the sigma-free surface region, inasmuch as it is presumed that sigma does not form in this grade of steel if initially balanced to be delta ferrite-free.

TABLE V.—X-RAY DIFFRACTION IDENTIFICATION OF MICRO-CONSTITUENTS IN STAINLESS STEELS.[a]

Type of Steel	Phases Observed After 10,000 hr at Indicated Temperature		
	900 F	1050 F	1200 F
17 Cr	ferrite, $Cr_{23}C_6$, Cr_2N(?)	ferrite, $Cr_{23}C_6$, sigma	ferrite, $Cr_{23}C_6$, sigma
18 Cr, 8 Ni (type 304)	austenite	austenite, $Cr_{23}C_6$	austenite, $Cr_{23}C_6$,
18 Cr, 8 Ni (0.03 C)	austenite, $Cr_{23}C_6$	austenite, $Cr_{23}C_6$	austenite, $Cr_{23}C_6$[b]
18 Cr, 8 Ni (Mo) (type 316)	austenite, $Cr_{23}C_6$	austenite, $Cr_{23}C_6$	austenite, $Cr_{23}C_6$
18 Cr, 8 Ni (Mo) (0.03 C)	austenite	austenite, $Cr_{23}C_6$	austenite, $Cr_{23}C_6$
18 Cr, 8 Ni (Mo, Cb)	austenite, CbC	austenite, CbC, $Cr_{23}C_6$	austenite, CbC, $Cr_{23}C_6$, sigma
18 Cr, 8 Ni (Ti) (type 321)	austenite, TiC, Cr_2N(?)	austenite, TiC, $Cr_{23}C_6$ (?), Cr_2N(?)	austenite, TiC, sigma[c]
18 Cr, 8 Ni (Cb) (type 347)	austenite, CbC	austenite, CbC, sigma	austenite, CbC, sigma

[a] Chromium K_α radiation. The micro-constituents were concentrated at sample surface by selective etching attack of the matrix with hydrochloric-picric acids in alcohol as described earlier (3).

[b] Also two faint lines at 2.997 and 2.19 Å; (these are not sigma).

[c] Sigma lines obliterate $Cr_{23}C_6$ and Cr_2N lines within range of diffraction angles examined.

subject to strain-aging embrittlement, with corresponding loss in notch-impact toughness at about 900 F, or other forms of embrittlement.

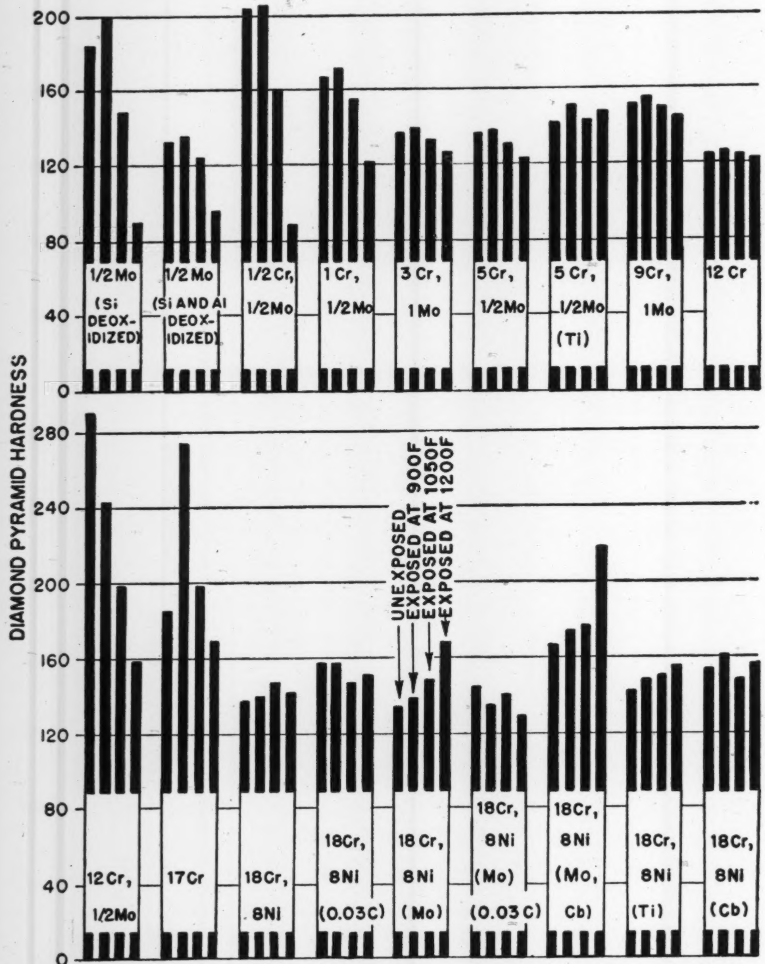


FIG. 2.—Effect of 10,000 hr Exposure at 900, 1050, or 1200 F on Hardness of Various Steels.

The fractured notch-impact specimens were employed for microstructural and hardness (Vickers-20 kg load) studies. These were made on sections normal to the notch and at positions not affected by the plastic deformation associated with fracture.

The microstructural examination of these steels was supplemented by X-ray diffraction examination to obtain positive proof of the identity of certain of the micro-constituents. For this purpose, certain micro-constituents were exposed at the surface of the sample by selective etching of the matrix with hydrochloric and picric acids in alcohol (overnight). Chromiun K_α radiation was employed in all instances, and all samples were given the same arbitrary etching treatment. The results are recorded in Table V.

It should be pointed out, in reference to Table V, that the procedure may not, for a number of reasons, have revealed all the phases which were present, but that if the diffraction lines of any phase were observed, there can be no doubt of the presence of that phase.

RESULTS AND DISCUSSION

The results are summarized in Fig. 1, which shows for each individual steel the effect of exposure on the hardness, microstructure, and temperature-variation of notch-impact strength. Table III lists the qualitative effect of exposure on the notch-impact strength; Tables IV and V give the nature of the observed microstructural changes and identify certain of the micro-constituents; and Fig. 2 summarizes all of the hardness results in bar-chart form.

Each of the points on the notch-impact curves in Fig. 1 represents the average of three tests, which, except in the "transition" range, generally agreed reasonably well with one another.[4] An exception was observed with the 5 Cr, $\frac{1}{2}$ Mo (Ti) steel which showed, in a number of samples, pronounced heterogeneity in grain size associated with the ingot segregation pattern. An arrow pointing upward on any point in Fig. 1 signifies that at least two of the test specimens did not break completely.

In reference to Table III, it should be made clear that only the general effect of the exposure with special reference to the transition temperature can be described in one word and that in some instances, for example, the notch-impact strength at the higher temperatures may actually be raised when the over-all effect is rated as an impairment.

Changes in Ferritic Steels and Their Effects:

The principal microstructural change observed in the ferritic steels, Fig. 1(*a*) to (*k*), and Table IV, was carbide spheroidization. In those alloys containing molybdenum and less than perhaps 3 per cent chromium, a precipitate, which seems to be characteristic of these steels, and which is generally associated with the presence of molybdenum (4), was also observed. Sigma phase was observed in the 17 Cr steel after exposure at 1050 F or 1200 F but not after exposure at 900 F; in this latter case the precipitate may have been too fine for detection. Essentially complete decarburization occurred in the steels of lowest alloy content exposed at 1200 F, and no doubt some decarburization also occurred in other instances.

These changes in microstructure caused changes in hardness and notch-impact strength. The two $\frac{1}{2}$ Mo steels, as well as the $\frac{1}{2}$ Cr, $\frac{1}{2}$ Mo and 1 Cr, $\frac{1}{2}$ Mo steels, hardened slightly on exposure at 900 F and then softened with increase of exposure temperature. The softening resulting from exposure at 1200 F is attributable to decarburization. The notch-impact strength of the first two steels was impaired by exposure at all temperatures studied, whereas the 1 Cr, $\frac{1}{2}$ Mo steel was impaired only at the two higher temperatures and remained essentially unaffected at 900 F. The $\frac{1}{2}$ Cr, $\frac{1}{2}$ Mo steel was impaired at 900 F, but improved at 1050 and 1200 F.

The next five steels in order of increasing alloy content were not greatly altered in hardness as a consequence of exposure, owing to their relatively coarse initial microstructures. The notch-impact strength was either unaffected or impaired, but was not improved in any instance.

The 12 Cr, $\frac{1}{2}$ Mo steel softened considerably and improved in notch-impact strength. This is attributable to the relatively high hardness of the initial microstructure which contains martensite.

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[4] The individual results of the notch-impact tests are too numerous to record here, but will be furnished by the authors upon request.

The 17 Cr steel was the only one to show substantial hardening. This occurred on exposure at 900 F and was associated with considerable impairment of the notch-impact strength, Fig 1(*k*). These changes are a manifestation of so-called 885 F embrittlement, which has long been known to occur in this and higher-chromium grades of ferritic steel. The causes of this embrittlement have never been established; one theory which attributes the phenomenon to the sigma phase receives some support, but not proof, from the presence of sigma after exposure of this steel at 1050 and 1200 F, which was confirmed by X-ray examination.

Changes in Austenitic Steels and Their Effects:

Two microstructural changes were observed in the austenitic steels, namely, carbide precipitation and sigma precipitation (Fig. 1(*l*) to (*r*) and Tables IV and V).

Of the changes observed in the several austenitic steels, the most interesting is that carbide occurs in those with a maximum of 0.03 per cent carbon. Until recently it has been widely assumed that this amount of carbon so slightly exceeds the solubility limit in austenite in the sensitization temperature range that carbide sensitization and its attendant ills are precluded. Yet the microstructural examination reveals the presence of a surprisingly large amount of a phase having the characteristics of carbide, and X-ray diffraction confirms its presence. Check chemical analysis after exposure showed that carbon content was the same as before exposure.

It is also noteworthy that no sigma was observed in these exposed low-carbon (0.03 max) steels except as a central longitudinal segregation in the plain 18 Cr, 8 Ni type, wherein a small amount of a massive phase having the etching characteristics of sigma was observed, corresponding with a similar segregation of a small quantity of delta ferrite in the unexposed metal. The presence of sigma could not, however, be confirmed by X-ray diffraction, owing presumably to an insufficient average concentration over the region irradiated by the X-ray beam. Since neither sigma nor delta ferrite was detected in the surface region, where the photomicrographs of Fig. 1(*m*) were taken, it is to be presumed that the presence of sigma in the central region is attributable to the delta ferrite initially there, and that if the steel is initially free of delta ferrite, no sigma will develop. Our failure to detect sigma phase, except as just stated, as well as the occurrence of a relatively large quantity of carbide in these steels is of interest in relation to the hypothesis recently advanced in a number of papers (but for which apparently only Binder and Brown (5) offer any experimental evidence) that the susceptibility of the 0.03 per cent carbon (max) grades containing molybdenum to intergranular corrosion under certain conditions is attributable to the presence of sigma.

In the austenitic steels with normal carbon, sigma phase was observed in the 18 Cr, 8 Ni (Mo, Cb); 18 Cr, 8 Ni (Ti); and 18 Cr, 8 Ni (Cb) grades after exposure at 1200 F where it was relatively massive in form, and in the last also at 1050 F, but as suggested earlier it may have been present in other instances and escaped detection. Thus, the low level of notch-impact strength of the 18 Cr, 8 Ni (Mo) grade after 10,000 hr at 1200 F parallels the trend (6) in a heat of the same grade in which sigma was definitely present after a 3000-hr creep test at 1300 F, and probably after a similar test at 1100 F, and suggests that sigma is probably present in the 1200 F series.

The hardness changes, Fig. 2, were of relatively small magnitude in all except the two molybdenum-containing grades of higher carbon content, in which hardness increased with increasing exposure temperature. In the case of the 18 Cr, 8 Ni (Mo, Cb) grade, the increase in hardness on exposure at 1200 F presumably is attributable to the presence of sigma; what causes it in the 18 Cr, 8 Ni (Mo) grade is not clear, although there may be a submicroscopic precipitation of sigma.

The changes in notch-impact strength with temperature are of interest. With minor exceptions, the notch-impact strength at room temperature decreased with increase of exposure temperature. This was especially pronounced in the two molybdenum-containing grades of higher carbon content. With decreasing temperature of the notch-impact test, further decrease of notch-impact strength occurred for most of the exposed materials with the notable exceptions of the 18 Cr, 8 Ni (0.03 C) and 18 Cr, 8 Ni (Cb) grades which remained substantially at their ambient-temperature level. Of the unexposed materials, only the 18 Cr, 8 Ni (Mo, Cb) and 18 Cr, 8 Ni (Ti) grades showed significant decrease in notch-impact strength with lowered temperature.

SUMMARY

The qualitative effects of the exposure on notch-impact strength shown in Fig. 1 are summarized in Table III. It will be noted that notch-impact strength was impaired in about two-thirds of the steel exposures; in another 25 per cent it was essentially unaffected, whereas in the remaining 10 per cent it was improved. In terms of exposure temperature, impairment occurred in 13 steels at 1050 F and also at 1200 F, and in 8 steels at 900 F. Improvement occurred in two steels at each of the exposure temperatures, whereas no change occurred in eight steels at 900 F but in only three at 1050 F and also at 1200 F. The two steels, which in terms of notch-impact strength were relatively unaffected by exposures at all three temperatures were the 9 Cr, 1 Mo and the 12 Cr (annealed).

The microstructural changes observed are summarized in Table IV. These were, of course, more advanced the higher the exposure temperature. In the ferritic steels, carbide spheroidized, and in some instances a finely dispersed precipitate of the type previously reported in molybdenum-containing steels (4) was observed. In the steels having the lowest alloy content, complete decarburization occurred during exposure at 1200 F. No graphitization was noted in any of the steels. In the austenitic steels, both carbides and sigma precipitated in the 18 Cr, 8 Ni, (Mo, Cb); 18 Cr, 8 Ni (Ti); and 18 Cr, 8 Ni (Cb) varieties. Sigma was also observed in the 17 Cr steel and in a segregated region of the 18 Cr, 8 Ni (0.03 C) steel. Also noteworthy was the discovery and identification of a carbide network in the grades containing 0.03 per cent max carbon.

The changes in hardness, noted in Fig. 1 and charted in Fig. 2, reflect the changes in microstructure for the most part. In general the low- and high-alloyed ferritic steels showed the greatest change in hardness, whereas in the intermediate ferritic steels and in the austenitic steels, with the exception of 18 Cr, 8 Ni (Mo, Cb) and 18 Cr, 8 Ni (Mo) the hardness changes were relatively minor.

It should be emphasized that the stability of any steel in a particular test depends not only upon its chemical composition but also upon its prior treatment.

Acknowledgments:

The steels used in this investigation were provided by the National Tube Co. and U. S. Steel Co. Duquesne Works, U. S. Steel Co., forged and heat treated the bars, and the exposures were made at Lorain Works of National Tube Co. E. J. Dulis gave valuable aid in the X-ray diffraction and microscopic examinations and W. L. Jensen assisted in making the notch-impact tests.

REFERENCES

(1) A. B. Wilder and J. D. Tyson, "Graphitization of Steel at Elevated Temperatures," *Transactions*, Am. Soc. Metals, Vol. 40, p. 233 (1948).

(2) A. B. Wilder and J. O. Light, "Stability of Steels at Elevated Temperatures," *Transactions*, Am. Soc. Metals, Vol. 41, pp. 141–166 (1949).

(3) E. J. Dulis and G. V. Smith, "Identification and Mode of Formation and Re-Solution of Sigma Phase in Austenitic Chromium-Nickel Steels," Am. Soc. Testing Mats., p. 3–37 (1951). (Symposium issued as separate publication, *STP No. 110.*)

(4) R. F. Miller, R. F. Campbell, R. H. Aborn and E. C. Wright, "Influence of Heat Treatment on Creep of Carbon-Molybdenum and Chromium-Molybdenum-Silicon Steels," *Transactions*, Am. Soc. Metals, Vol. 26, pp. 81–101 (1938).

(5) W. O. Binder and C. M. Brown, "Influence of Carbon and Molybdenum on the Intergranular Corrosion Resistance of Austenitic Chromiun-Nickel Steels with and without Columbium," Am. Soc. Testing Mats., p. 146 (1950). (Symposium issued as separate publication, *STP No. 93.*)

(6) G. V. Smith, E. J. Dulis and E. G. Houston, "Creep and Rupture of Several Chromium-Nickel Austenitic Stainless Steels," *Transactions*, Am. Soc. Metals, Vol. 42, pp. 935–980 (1950).

DISCUSSION

MR. J. J. HELLER.[1]—I should like to ask the authors about the precipitate generally observed in low-alloy molybdenum-bearing materials. I believe there is an unidentified, groundmass precipitate observed generally in these steels, which, under certain conditions, tends to agglomerate and form chains within the ferrite grains.

I wonder whether the authors observed any such change.

MR. W. B. HOYT.[2]—I should like to inquire about the presence of sigma phase as shown in the 17 per cent chromium steel. Apparently, in that particular case it did not reduce the impact strength to the extent it did in the molybdenum-bearing austenitic steels. Can the authors give a theory as to why it behaved that way?

MR. M. A. SCHEIL[3] (*by letter*).—The authors are to be congratulated for a very fine piece of work on the structural instability of a number of high-temperature steels.

Of extreme interest to the writer are the comments regarding the intergranular precipitation observed in the austenitic 18-8 and 18-8 molybdenum 0.03 per cent carbon max stainless steels when heated between 900 and 1200 F. This finding is of great importance and the writer would be extremely interested in whether the authors subjected any of the steels showing the chromium carbide phase to a boiling 1:8 copper sulfate sulfuric acid corrosion test. The writer and Mr. Ebling have discussed in a previous paper, "Some Observations on Tests for Intergranular Susceptibility of 18-8 Mo Stainless Steels,"[4] that a precipitate formed after 800 and 900 F sensitization for 500 hr in 18-8 molybdenum steel 0.03 per cent carbon max could not be entirely carbide inasmuch as there was no indication of intergranular attack with the boiling copper sulfate sulfuric acid test. However in the boiling nitric acid test which does attack sigma and carbide as well, there can be considerable attack after long-time low-temperature heating. We believe that the authors should check these low-carbon grades of 18-8 subjected to the long-time heating at 900, 1050, and 1200 F with the boiling 65 per cent nitric, nitric-hydrofluoric, and 1:8 copper sulfate sulfuric acid corrosion tests. A knowledge of the corrosion behavior will help further to identify the structure. It could well happen that the 0.03 per cent carbon max austenitic steels subjected to the 1200 F exposure would not be intergranularly attacked.

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[2] Materials Engineer, The M. W. Kellogg Co., Jersey City, N. J.

[3] Director of Metallurgical Research, A. O. Smith Corp., Milwaukee, Wis.

The writer is extremely interested in the treatment of the data for the 10,000 hr exposure tests exhibited in Fig. 1. The low-temperature notch toughness after 10,000 hr at 1200 F exposure for the 18-8 molybdenum Type 316 steel would be suspected by some of the experience which we have had on 18-8 steels in a highly sensitized condition when tested at temperatures below −150 F. It would be interesting to know at what point on the transition temperature curve for steel (*n*) the Charpy fracture went from transgranular to intergranular. This refers, of course, to the specimens tested after exposure at 1200 and 1050 F.

The writer was extremely interested in the work reported on the 9 per cent chromium and 12 per cent chromium steels, since somewhat similar work as reported by the authors and by Wilder and Light[5] is being performed by the writer's company covering a series of seven 12 per cent chromium steels subject to several years' exposure at 750 and 900 F. The test specimens in the writer's program cover three conditions of heat treatment:

(*a*) Annealed at 1550 F, furnace cooled.

(*b*) Annealed and cold worked an average of 13 per cent.

(*c*) Grain coarsened at 2000 F, furnace cooled.

Test specimens after exposure cover the tensile properties of unstressed specimens and the notch impact strength using standard keyhole notch Charpy specimens.

In the exposure which has progressed to approximately 13,000 hr to date at 750 F and 9000 hr at 900 F the following results are of interest in comparison to quite similar analysis steels reported by Wilder and Light and by the authors.

In Fig. 1, steel (*h*), the 9 per cent chromium-1 per cent molybdenum steel reported by the authors appears to be affected by the exposure at 1050 F as the impact strength is approximately 20 ft-lb lower than the unexposed material. This does not seem to agree with the authors' conclusions stated in Table III for this material.

Of interest are the writer's tests on a steel containing 0.10 per cent carbon

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[4] Symposium on Evaluation Tests for Stainless Steels, Am. Soc. Testing Mats. p. 121 (1950). (Issued as separate publication *STP No. 93.*)

[5] A. B. Wilder and J. C. Light, "The Stability of Steels at Elevated Temperatures," *Transactions*, Am. Soc. Metals, Vol. 43, pp. 141–166 (1949).

8.16 per cent chromium and 0.92 per cent molybdenum. This material in the coarsened condition at 2000 F followed by 1250 F temper and then subjected to exposure showed the following: In the unexposed condition the Charpy value averaged 39.5 ft-lb. After 750 F the impact value was considerably decreased. Three values taken showed 20.4 ft-lb, 6.6 ft-lb and 40.6 ft-lb for an average of 22.5 ft-lb. After 900 F the material averaged 37.2 ft-lb. Our results at 900 F are in agreement with the authors' conclusions. However, it should be added that in private correspondence with Mr. H. D. Newell it has been noted in his experience that there had been a few cases of temper embrittlement in both 5 per cent chromium-$\frac{1}{2}$ per cent molybdenum steel and also in 9 per cent chromium-1 per cent molybdenum steel where these materials have been grain coarsened in hot working operations without suitable rectification of the grain structure on annealing. Normally this does not happen on properly processed material either on long heating or slow cooling through the so-called temper embrittlement range.

TABLE VI.—AVERAGE OF THREE STANDARD KEYHOLE NOTCH CHARPY TESTS, FT-LB.

	Annealed	Annealed and Cold Worked	Grain Coarsened
Unexposed	38.9	41.9	34.6
After 750 F	35.6	30.0	23.2
After 900 F	41.2	39.1	23.8

The authors' 12 per cent chromium-$\frac{1}{2}$ per cent molybdenum steel is the same as reported by Wilder and Light.[5] Both of these papers report an increase in the notch toughness of this steel after 900, 1050 and 1200 F for 10,000 hr.

The writer used a steel containing 0.13 per cent carbon, 12 per cent chromium and 0.54 per cent molybdenum. This steel, when tested in the three conditions, showed a decrease in all cases from the unexposed material at the 750 F exposure. When the material was tested in the coarsened condition at 2000 F our exposure at 900 F also showed about the same decrease in notch toughness as the 750 F exposure.

The accompanying Table VI shows the notch impact strength of the tested material.

Thus our results, particularly at 750 F, disagree with the authors' conclusions for 12 per cent chromium-$\frac{1}{2}$ per cent molybdenum steel.

Of particular interest were the authors' results on standard type 410 (12 per cent chromium) and those reported by Wilder and Light, namely, their types 410 and 416. Both of the type 410 analyses were below 13 per cent chromium and showed no decrease in notch toughness after exposure at 900 and 1050 F. These results are in line with several exposure tests made by the writer, and the writer finds no decrease in notch toughness when type 410 steels with a 13.0 per cent chromium max are tested after exposure at 750 F.

Of interest, however, are the results reported for a type 416 analysis by Wilder and Light. This steel contained 13.39 per cent chromium and 0.11 per cent carbon. When tested after exposure at 900 F and 1050 F for 10,000 hr, it showed a decided decrease in notch toughness. This is in line with the work done by the writer and indicates that a restricted analysis range for the types 410 and 405 steels as regards chromium, columbium, titanium, and carbon must be considered if these valuable 12 per cent chromium steels are to retain their toughness and ductility after exposure in the temperature range of 700 to 1000 F.

It should not be construed that the steels that the writer speaks of as grain coarsened at 2000 F received extraordinary coarse grain by this treatment. The reason for using this heat treatment was found by experience, namely, that the 12 per cent chromium steels when heated for forming should not exceed a maximum temperature of 2000 F if the material is to be used in the so-called 885 F embrittlement temperature range. Most all of the 12 per cent chromium steels, even those modified by molybdenum and aluminum, will not show undue coarsening at temperatures below 2000 F, but when these are heated to 2100 F or higher, they will develop extremely coarse grains and this structure then becomes very susceptible to strain embrittlement and aging embrittlement.

In connection with the possible formation of sigma phase in the 12 per cent chromium steels, the authors might like to consider the method used by J. J. Heger as reported in the "Symposium on the Nature, Occurrence, and Effects of Sigma Phase,"[6] wherein cold work and extended heating periods of 900 to 1200 F were used to precipitate a phase which was identifiable, and in the 17 per cent chromium analysis tested by Heger was positively identified as sigma at 1050 F.

MR. G. V. SMITH (*authors' closure*).—In answer to Mr. Heller, we did observe, as described in our paper, the groundmass precipitate often found in low-alloy molybdenum-containing steels used for service at elevated temperatures. We cannot say, however, whether this phase actually agglomerates under certain conditions, as Mr. Heller suggests, or whether the agglomerate is actually another phase.

Mr. Hoyt has inquired about the effect of sigma in the 17 per cent chromium steel. Although sigma, when formed at 1200 F, did not impair the notch impact strength of the 17 per cent chromium steel, severe impairment was observed in this steel when the sigma was formed at 1050 F. (Even greater impairment occurred on exposure at 900 F; no sigma was observed, but it may have been present, and escaped detection owing to fineness of dispersion). Thus, it would appear that sigma can be just as embrittling in the ferritic chromium steels as in the austenitic chromium-nickel steels. The specific effect for any set of conditions, for example, exposure temperature, must, however, depend upon the specific distribution, as well as amount, of the sigma phase, and also, of course, of any other phases present.

In reply to Mr. Scheil, the exposed austenitic stainless steels have been subjected to both nitric acid and copper sulfate-sulfhuric acid corrosion tests. These tests were arranged by J. J. Heger and were made at South Works, United States Steel Company, by C. S. Walton, to both of whom we are grateful for permission to summarize here the results.

The test samples were taken from broken notch impact test specimens. They were approximately $\frac{5}{8}$ by $\frac{3}{8}$ in. cut from half impact specimens after the fractured section had been removed; a $\frac{1}{4}$-in. hole was drilled for suspension. Each sample was hand finished on 120 grit abrasive to insure a uniform surface.

For the nitric acid tests, the samples were immersed in boiling 65 per cent nitric acid for five 48-hr periods and the weight losses were converted into corrosion rates expressed as inches penetration per month (ASTM Recommended Practice A 262 – 44 T).[7] If the corrosion rate for any boiling period, after the first, exceeded 0.005 inches per month, the test was discontinued.

For the copper sulfate-sulfuric acid tests the samples were exposed to a boiling solution of 13 grams Cu

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[6] J. J. Heger, "The Formation of Sigma Phase in 17 Per Cent Chromium Steel," Symposium on The Nature, Occurrence, and Effects of Sigma Phase, Am. Soc. Testing Mats., p. 75 (1951). (Symposium issued as a separate publication *STP No. 110*).

[7] Tentative Recommended Practice for Boiling Nitric Acid Test for Corrosion-Resisting Steels (A 262 – 44 T), 1949 Book of ASTM Standards, Part 1, p. 874.

$SO_4 \cdot 5H_2O$, 47 ml H_2SO_4 (specific gravity 1.84) and approximately 950 ml H_2O for 500 hr. A minimum of 35 ml of solution per square inch of surface area was used. At approximately 100-hr intervals, water was added to the solution to maintain constant volume. Because of the size and shape of the samples, the corrosion resistance in acidified copper sulfate solution was evaluated by weight less measurements and microscopic examination rather than by the more usual method of observation of cracking in bend test.

The individual results are too numerous to record here, but Table VII provides a summary of the observations. In the unexposed state, all grades proved satisfactory in both the acidified copper sulfate and boiling nitric acid tests. In respect to the latter test, corrosion resistance is considered satisfactory when the average penetration rate for five 48-hr boiling periods is less than 0.002 in. per month, a commonly accepted criterion. After 10,000-hr exposure at 900 F, types 304 and 304(0.03 C) showed respectively severe and slight intergranular attack in the $CuSO_4H_2SO_4$ test, whereas the remaining grades showed no such attack. In the boiling nitric acid tests only types 316 Cb and 347, that is, the two columbium-containing grades, showed a penetration rate less than 0.002 in. per month.

TABLE VII.—SUMMARY OF RESULTS OF CORROSION TESTS OF AUSTENITIC STAINLESS STEELS.

Type	Unexposed		Exposed at 900 F		Exposed at 1050 F		Exposed at 1200 F	
	65 per cent Nitric Acid Test[a]	Acidified Copper Sulfate Test[b]	65 per cent Nitric Acid Test[a]	Acidified Copper Sulfate Test[b]	65 per cent Nitric Acid Test[a]	Acidified Copper Sulfate Test[b]	65 per cent Nitric Acid Test[a]	Acidified Copper Sulfate Test[b]
304	0.00071	None	0.07506	Severe	0.04785	Severe	0.00110	None
304(0.03C)	0.00062	None	0.05750	Slight	0.00376	None	0.00075	None
316	0.00098	None	0.01645	None	0.16290	Severe	0.00650	Slight
316(0.03C)	0.00096	None	0.00317	None	0.13070	Severe	0.00346	None
316 Cb	0.00053	None	0.00103	None	0.03702	Slight	0.00544	None
347	0.00089	None	0.00158	None	0.00361	None	0.00141	None
321	0.00151	None	0.00218	None	0.00479	None	0.00262	None

[a] Average penetration rate—inch per month.
[b] Based on microscopic examination of extent of intergranular attack.

All of the grades, when exposed for 10,000 hr at 1050 F, failed to pass the boiling nitric acid test, and only types 304(0.03 C), 347 and 321 failed to show intergranular attack in the $CuSO_4$-H_2SO_4 test. After exposure at 1200 F, all grades were free of intergranular attack in the $CuSO_4$-H_2SO_4 test except type 316, but only types 304, 304(0.03 C), and 347 showed less than 0.002 in. per month penetration. In all grades exposed at 1200 F, however, the penetration rate was considerably less than that observed after 1050 F exposure. This improvement for the higher exposure temperature is doubtless attributable to the breaking up and agglomeration of the precipitate phases, which tended to form continuous grain boundary networks at lower temperature. It is also noteworthy that the relatively large amounts of sigma observed in some steels after exposure at 1200 F were not especially deleterious to the corrosion resistance, as a consequence no doubt, of their relatively massive form. These observations on corrosion resistance would appear thus to substantiate further the conclusions of the paper regarding the occurrence of carbide in the low carbon (0.03) grades of 304 and 316.

Mr. Scheil has also inquired about the temperature at which the fracture of steel N (type 316) when exposed at 1050 or 1200 F changed from transgranular to intergranular. We have not examined the fractures from this standpoint, and whereas we doubt that, in general, notch impact test fractures are intergranular at low temperature, it is possible that they are so in some specific cases such as when a brittle grain-boundary network exists. We hope to find time at some future date to explore this question in some detail. We can report at this time that the macroscopically observable change from "fibrous" to "granular" (which is not necessarily synonomous with intergranular) appearance occurred in this steel at about −100 F when exposed at 1050 F and at about room temperature when exposed at 1200 F. For either the unexposed metal or that exposed at 900 F, no "granular" fracture was observed.

We were interested to learn the preliminary results of Mr. Scheil's long time exposure experiments on 12 per cent chromium steels at 750 and 900 F, and shall look forward to his forthcoming paper. Whereas it is true, as Mr. Scheil states, that the impact strength of the 9 Cr-1 Mo steel has been lowered somewhat by exposure at 1050 F, this is true only at temperatures above the transition temperature. Our Table III, however, is based on "the general effect of the exposure with special reference to the transition temperature," and *on this basis*, as stated in the paper, our conclusion is still correct that the notch impact strength of 9 Cr-1 Mo steel is unimpaired by exposure at 1050 F. We would agree, however, as we also stated in the paper, to the shortcomings of attempting a one-word description of the behavior such as we used, but nevertheless thought such an evaluation desirable.

In further reply to Mr. Scheil, we do not consider that he is warranted in stating that his results for 12 Cr-$\frac{1}{2}$ Mo steel "disagree" with our conclusions, since we did not expose samples at 750 F nor study the material in other than the initial condition. Within the area of duplication, the results do not appear to be significantly different.

NOTCH TOUGHNESS OF FOUR ALLOY STEELS AT LOW TEMPERATURES*

By W. B. Seens,[1] W. L. Jensen,[1] and O. O. Miller[1]

Synopsis

This paper presents data on the effect of temperature in the range from room temperature to −315 F on notch-toughness by the Charpy keyhole test of three low-alloy steels and one medium-alloy steel intended for use at low temperatures. Steels containing nominally 2.5 per cent nickel ("2215"),[2] 3.5 per cent nickel (2317), 3.5 per cent nickel and 0.25 per cent molybdenum (4815) and 9 per cent nickel (2810) were investigated. Charpy impact specimens with keyhole notch were taken from ½-in. thick plates and from 2-in. thick plates of all four steels and from 0.24-in. or 0.30-in. wall tubing of 2317 and 2810 steels.

For the plates, considerable difference in energy was found between longitudinal and transverse specimens, except at low temperatures where the energy absorbed was small. Little difference was found between specimens from two different locations in the plate. The tests showed that notch-toughness at low temperature could be greatly improved by heat treatment, the degree of improvement depending on method of heat treatment, hardenability of the steel, and plate thickness.

The impact specimens from the tubing were sub-size and therefore absorbed less energy at the higher temperatures than the standard specimens from the plate. However, the tubing retained a greater proportion of its toughness at low temperatures, due both to the faster cooling rate associated with thinner material and to the less severe restraint in the test specimens.

Because of the increasing number of low-temperature applications, there is considerable demand for data on mechanical properties of steel at temperatures well below atmospheric. In many such applications, notch-toughness is of particular interest because at a sufficiently low temperature all steels except the austenitic stainless grades are known to behave in a brittle manner, particularly in the notched condition. The present investigation was undertaken to provide such information on notch-toughness for several low-alloy and medium-alloy steels of commercial interest for low-temperature service.

Material

Four grades of steel, containing approximately 2.25 per cent nickel, 3.4 per cent nickel, 3.3 per cent nickel together with 0.25 per cent molybdenum, and 9 per cent nickel, were investigated. Composition of these steels, corresponding respectively to AISI-SAE grades "2215",[2] 2317, 4815, and 2810, is given in Table I.

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* Presented at the Fifty-fourth Annual Meeting of the Society, June 18–22, 1951.

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[2] The designation "2215" is used for convenience, even though this steel is not an accepted SAE or AISI grade.

TABLE I.—COMPOSITION OF STEELS.

Grade	Product	Chemical Composition,[a] per cent (Check Analysis)							Corresponding ASTM Spec.
		C	Mn	P	S	Si	Ni	Mo	
"2215"......	Plate	0.16	0.53	0.018	0.039	0.24	2.34	0.01	A 203 - 49 T Grade A
2317........	Plate	0.13	0.45	0.012	0.038	0.22	3.37	Nil	A 203 - 49 T Grade D
2317........	Tubing	0.17	0.50	0.010	0.022	0.29	3.45	0.04	...
4815........	Plate	0.15	0.53	0.019	0.043	0.26	3.32	0.25	...
2810........	Plate and Tubing	0.10	0.50	0.007	0.013	0.26	9.02	Nil	...

[a] All steels were made by fine-grain practice.

TABLE II.—HEAT TREATMENT AND MECHANICAL PROPERTIES OF PLATES.

Grade	Plate Thickness, in.	Heat Treatment	Hardness, BHN	Tensile Properties[a]				
				Tensile Strength, psi	Yield Strength, psi	Elongation per cent		Reduction of Area, per cent
						in 2 in.	in 8 in.	
"2215"	0.5	Normalized 1600 F	149	73 500	53 700	...	25.5	...
"2215"	0.5	Normalized 1600 F, Tempered 1100 F	156	73 700	53 500	...	28.0	...
"2215"	0.5	Quenched from 1525 F, Tempered 1100 F	156	74 600	53 100	...	24.5	...
"2215"	2.0	Normalized 1600 F	137	70 900	48 400	33.5	...	66.0
"2215"	2.0	Normalized 1600 F, Tempered 1100 F	140	70 700	51 000	34.0	...	65.9
2317	0.5	As Hot Rolled	152	72 800	48 600	...	24.5	...
2317	0.5	Normalized 1600 F	152	73 500	56 000	...	26.0	...
2317	0.5	Normalized 1600 F, Tempered 1100 F	156	73 100	56 100	...	27.0	...
2317	0.5	Quenched from 1525 F, Tempered 1100 F	187	85 500	70 900	...	17.5	...
2317	2.0	As Rolled	131	65 500	37 400	33.0	...	63.1
2317	2.0	Normalized 1600 F	143	71 200	49 800	35.5	...	65.8
2317	2.0	Normalized 1600 F, Tempered 1100 F	146	71 300	52 400	35.5	...	67.3
4815	0.5	Normalized 1600 F	201	91 900	72 900	...	22.0	...
4815	0.5	Normalized 1600 F, Tempered 1100 F	192	87 000	71 000	...	21.5	...
4815	0.5	Quenched from 1525 F, Tempered 1100 F	217	99 600	84 100	...	12.0	...
4815	2.0	Normalized 1600 F	192	89 200	63 200	27.0	...	65.9
4815	2.0	Normalized 1600 F, Tempered 1100 F	183	84 100	65 700	29.5	...	68.1
4815	2.0	Quenched from 1525 F, Tempered 1100 F	207	94 900	79 200	27.0	...	70.8
2810	0.5	Double Normalized, 1650 and 1450 F, Tempered 1050 F	229	104 500	81 000	...	17.5	...
2810	0.5	Quenched from 1450 F, Tempered 1050 F	241	104 400	97 000	...	17.5	...
2810	2.0	Double Normalized, 1650 and 1450 F, Tempered 1050 F	229	104 100	79 000	28.0	...	66.8
2810	2.0	Quenched from 1450 F, Tempered 1050 F	241	106 100	99 500	26.5	...	73.4

[a] Longitudinal specimens; standard 8 in. GL plate specimens from 0.5 in. plate and standard 0.505 in. diam, 2 in. GL specimens from 2 in. plate.

[b] Quenching from 1525 F or 1450 F was in water. All tempered specimens were quenched after tempering.

Tempering period 1½ hr except

2215—2 in., 2315—2 in., 4815—2 in.—N	3 hr
4815—2 in.—Q, 2810—2 in.—N	4 hr
2810—2 in.—Q	5 hr

All of these steels were made by "fine-grain" deoxidation practice. The "2215" steel is within the composition limits for grade A, and the 2317 steel within the limits for grade D of ASTM Tentative Specifications for Nickel-Steel Plates for Boilers and Other Pressure Vessels (A 203 - 49 T).[3] A higher hardenability steel, 4815, was included because it contains about the same amount of nickel as 2317, yet can be hardened in heavier sections, or for the same section by less drastic cooling. The 9 per cent nickel steel (**1, 2, 3**)[4] was included because of its apparently favorable low-temperature properties.

Plates of all four steels in two thicknesses—½ in. and 2 in.—were rolled straight-away (no cross-rolling) to obtain the maximum amount of directionality in mechanical properties. Sections of these plates, representing the full thickness of the plate, were heat treated under commercial conditions by the methods listed in Table II, which also gives the resulting hardness and tensile properties. The heat treatments included normalizing, normalizing and tempering, and quenching and tempering, thus making it possible to compare the effect of heat treatment for these grades and sizes of plate. The 2317 plates were also tested in the hot-rolled condition. For the lower hardenability steels, "2215" and 2317, only the ½-in. plates were quenched and tempered. For 2810, the plates were double-normalized and tempered at 1050 F in accordance with recommended practice (2), or were quenched and tempered.

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[3] **1949 Book of ASTM Standards, Part 1, p. 382.**

[4] **The boldface numbers in parentheses refer to the list of references appended to this paper see p. 928.**

Tensile properties of "2215" plate after heat treatment are within the limits for grade A in ASTM Specification A 203. Properties of the heat-treated 2317 plate likewise conform to those for grade D of this specification, except in the quenched-and-tempered condition where the strength is somewhat higher and the elongation slightly lower than specified. Both 4815 and 2810, in all conditions tested, are considerably stronger than required for any grade in Specification A 203.

Sections of 2317 tubing having 4.5-in. outside diameter by 0.237-in. wall and of 2810 tubing having 3.5-in. outside diameter by 0.30-in. wall were heat treated as shown in Table III. Hardness of 2317 tubing is considerably higher than that of the plate heat treated comparably, partly because of the difference in carbon content and partly because of the difference in size of section.

SAMPLING AND TESTING PROCEDURE

Full-size Charpy impact specimens from the plate and half-size specimens from the tubing, both with keyhole notch, were used. For the plate, both longitudinal specimens and transverse specimens were tested, whereas only longitudinal specimens could be obtained from the tubing. In all instances, the axis of the notch was perpendicular to the surface of the plate or tube.

All plates were sampled at a location midway between the edge and centerline. In addition, samples representing the centerline location in 2317 and 2810 plates for each of the heat treatments were tested in order to determine whether location in the plate had an appreciable effect on notch-toughness. All specimens represented material midway between the two surfaces of ½-in. plate or tubing and midway between surface and center 2-in. plate.

TABLE III.—HEAT TREATMENT AND HARDNESS OF TUBING

Grade	Size, in.	Heat Treatment[a]	Hardness BHN
2317	4.5 OD × 0.237 wall	Normalized 1550 F	187
2317	4.5 OD × 0.237 wall	Normalized 1550 F, Tempered 1150 F	179
2317	4.5 OD × 0.237 wall	Quenched from 1525 F, Tempered 1150 F	207
2810	3.5 OD × 0.30 wall	Double Normalized 1650 and 1450 F, Tempered 1050 F	235
2810	3.5 OD × 0.30 wall	Quenched from 1450 F, Tempered 1050 F	255

[a] Quenched in water from 1525 or 1450 F; air cooled after tempering 1½ hr.

A Riehle machine of 220 ft-lb capacity, with striking velocity of 18.1 ft per sec, 2-mm anvil and striking edge radii, and 44-mm initial anvil span (40-mm clearance span) was used for the tests. This machine is equipped with a positive centering device which permits rapid and accurate positioning of the specimen.

Each group of specimens for testing at any one temperature was held at least 15 min in a bath at a slightly lower temperature, such that at the instant of breaking after a constant transfer interval of 5 sec the specimen was at the desired temperature. An alcohol-acetone bath cooled with dry ice was used for temperatures to −100 F, and non-flammable liquefied Freon 12 or 13 cooled with liquid nitrogen was used for lower temperatures.

Tests were made, in triplicate, at several temperatures over a range that included the transition from tough to brittle behavior; temperature intervals within the transition range were usually 25 F. Testing was continued until a temperature was reached at which all three specimens broke at a low energy level and with essentially 100 per cent granular fracture, unless the number of specimens available (21 to 24) was insufficient or the required test temperature was below −315 F, the lowest attainable with liquid nitrogen.

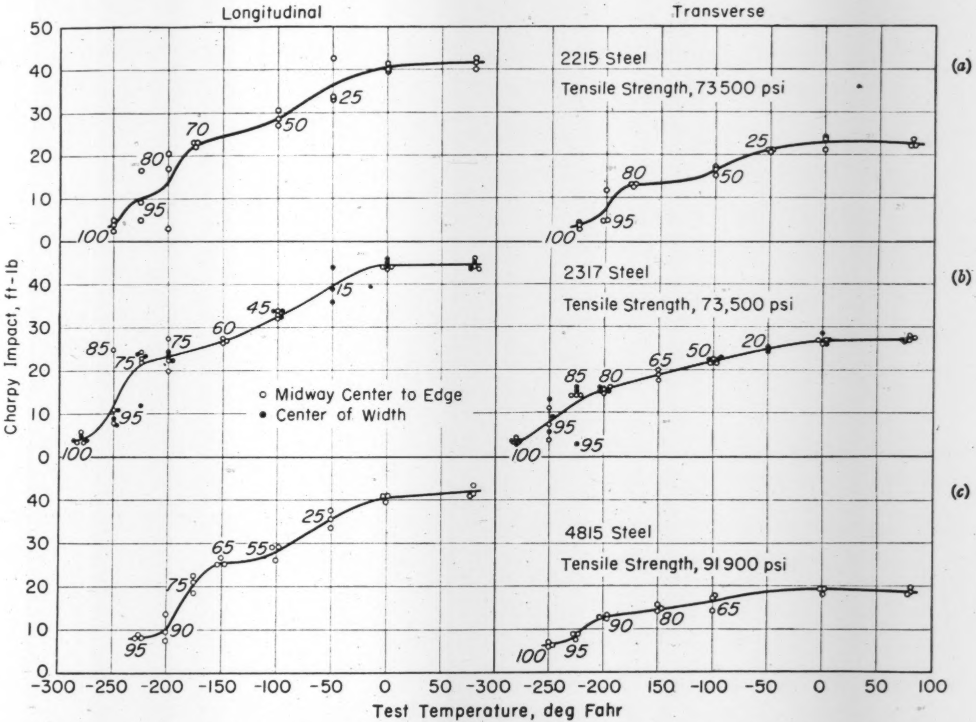

FIG. 1.—Effect of Temperature on Keyhole-Notch-Toughness of Normalized ½-in. Plate. (Numbers on chart represent percentage of granular fracture)

The area of the fracture having a granular appearance was measured with a binocular microscope, and the percentage was determined from tables relating this area to the original cross-sectional area under the notch.

RESULTS FOR PLATES

Effect of Test Temperature:

Notch-toughness of all the steels tested, except the 2810 grade, decreased gradually at first, then more abruptly as the test temperature was lowered. The drop in toughness was accompanied, as usual, by an increase in the proportion of the fracture that appeared granular. These changes are illustrated in Fig. 1 for "2215," 2317, and 4815, in the normalized condition. Notch-toughness of 2810 decreased slightly as the test temperature was decreased down to −315 F, as illustrated in Fig. 2. Evidently, a still lower temperature would be required to reduce the notch-toughness of this steel, measured in this manner, to a low value.

Frequently, the effect of temperature on notch-toughness is evaluated in terms of a "transition temperature" or "transition temperature range" (4, 5). The general significance of such a designation is clear but its evaluation is difficult and apt to be arbitrary. One reason for this is that the transition, or change, from "tough" to "brittle" behavior may occur over a fairly wide temperature range, without a clearly indicated beginning and end. Another reason is that the transition depends on the type of test and on whether energy absorbed or type of fracture is used as a criterion of toughness. Because of the arbitrary nature of any "transition temperature" that might be selected, the results of this investigation are presented in the form of curves relating energy absorbed in the Charpy test to temperature of test. In Figs. 1 and 2 the data for individual tests as well as average curves are shown. It is not feasible, however, to present all of the data in such detail and in many subsequent illustrations only the average curves are shown. In general, these average curves represent the original data as well as do those in Figs. 1 and 2.

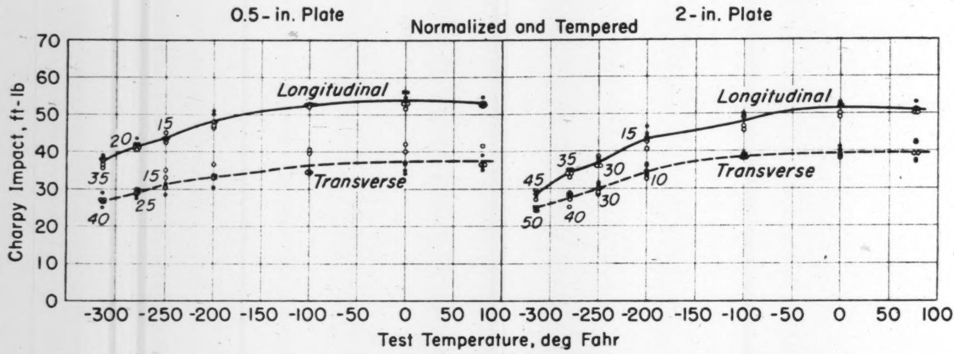

FIG. 2.—Effect of Temperature on Keyhole-Notch-Toughness of Normalized and Tempered 2810 Steel Plate.

½-in. plate, Tensile strength 104,500 psi.
2-in. plate, Tensile strength 104,100 psi.
(Numbers on chart represent percentage of granular fracture)

Steels "2215" and 2317 fulfill the requirements of Class 2 and Class 3, respectively, of ASTM Tentative Specification A 300 – 50 T[5] covering steel plates for pressure vessels to be used at low temperatures. According to this specification, the plates are to be normalized and their average Charpy value, based on three tests, is to be 15 ft-lb minimum at −75 F for steels similar to "2215" and 15 ft-lb minimum at −150 F for the 2317 grade. The particular heats tested are well above the minimum value at the respective specified test temperatures, as shown in Figs. 1 (*a*) and (*b*).

Effect of Location in Plate:

No significant difference in results was found between specimens from the centerline location and those from a location midway between center and edge of the plate. This is illustrated in Fig. 1(*b*) for 2317 steel and in Fig. 2 for 2810 steel. Data for other plate sizes and other heat treatments of these same steels, not reproduced here, confirm this conclusion. It is evident, therefore, that notch-toughness of these plates was fairly uniform, at least over the central half of the plate.

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[5] Tentative Specification for Steel Plates for Pressure Vessels for Service at Low Temperatures (A 300 – 50 T), 1950 Supplement to Book of ASTM Standards, p. 118.

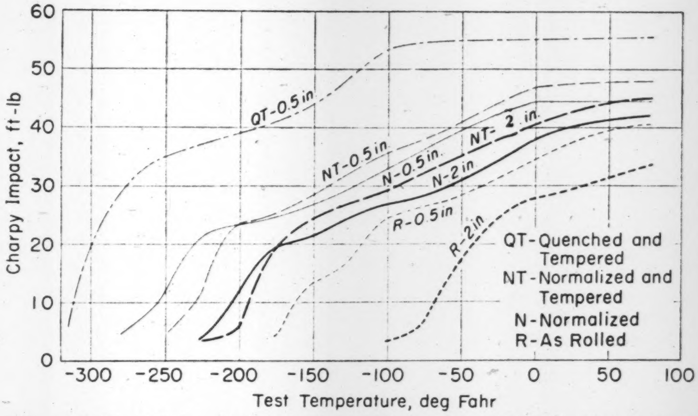


FIG. 3.—Effect of Heat Treatment and Plate Thickness on Keyhole-Notch-Toughness of 2317 Steel Plate. Longitudinal Specimens.

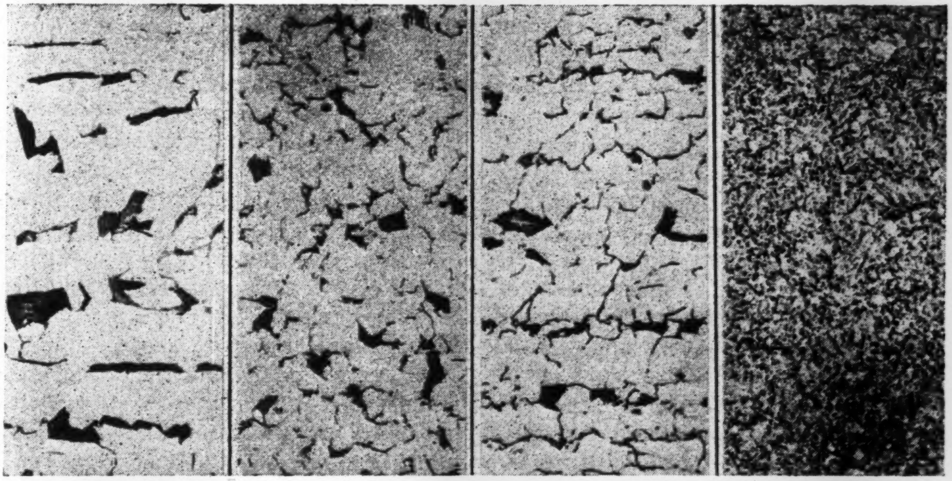

(*a*) (*b*) (*c*) (*d*)

FIG. 4.—Effect of Heat Treatment on Microstructure of ½-in. Plate of 2317 Steel (Original magnification × 500, reduced 20% in reproduction).
(*a*) Hot rolled. 15 ft-lb, average, at −140 F.
(*b*) Normalized. 15 ft-lb, average, at −240 F.
(*c*) Normalized, tempered. 15 ft-lb, average, at −220 F.
(*d*) Quenched, tempered. 15 ft-lb, average, at −310 F.

Comparison of Longitudinal and Transverse Specimens:

In general, all of the longitudinal specimens absorbed considerably more energy than the corresponding transverse specimens. As shown in Fig. 1, this difference was not observed at low temperatures where both were brittle, but at higher temperatures it was very marked in three of the steels. The difference was somewhat less in 2810 plate, although in this steel also, the curves for transverse specimens lie considerably below the corresponding ones for longitudinal specimens, as illustrated in Fig. 2. The differences shown in Figs. 1 and 2 are representative of those for the same steels heat treated in other ways, or for 2-in. plates of the three lower-alloy steels. These large differences are undoubtedly due in part the fact that all of the plates were hot rolled in one direction only. This was done deliberately in order to develop maximum directionality in properties. Differences between longitudinal and transverse properties would probably be less in cross rolled plates.

Effect of Heat Treatment:

Considerable improvement in notch-toughness at low temperature can be effected by proper heat treatment. An example of the magnitude of this improvement is shown in Fig. 3, representing the effect of normalizing, normalizing and tempering, and quenching and tempering ½-in. plate of 2317 steel. The effect of normalizing or normalizing and tempering the 2-in. plate also is shown, but the 2-in. plate was not quenched and tempered because this steel lacks sufficient hardenability to be hardened effectively in this size.

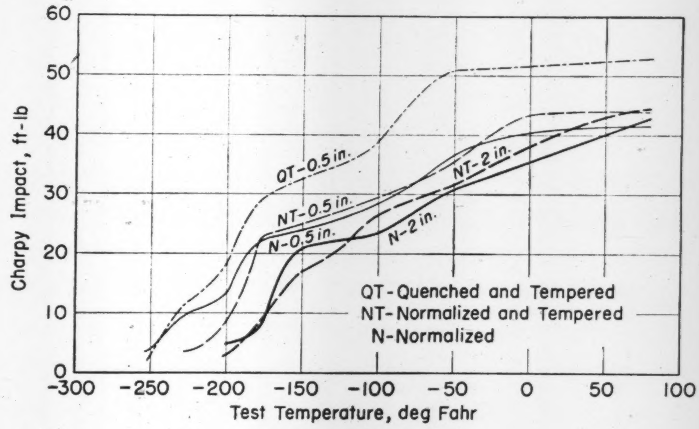
Considering first the ½-in. thick plate (thinner lines in Fig. 3), normalizing the hot-rolled plate shifted the absorbed energy curve to the left by a substantial amount, roughly 100 F over most of the range. Tempering this steel after normalizing had little effect; the upper portion of the curve was shifted slightly to the left and the lower portion slightly to the right. Quenching and tempering, however, greatly improved the notch-toughness, shifting the curve upward and to the left by a substantial amount despite the increased hardness and strength resulting from this treatment (Table II). The effect of normalizing or normalizing and tempering the 2-in. thick plate was about the same as for the ½-in. plate.

The effect of heat treatment on notch-toughness, illustrated in Fig. 5, is directly related to the change in microstructure of the steel. Microstructure of the ½-in. plate of 2317 is shown in Fig. 4. Normalizing the hot-rolled plate resulted in a somewhat finer ferrite grain size and more uniformly dispersed pearlite or carbide particles, both factors probably contributing to the improvement in notch toughness. Tempering this normalized steel did not appreciably affect its structure, nor its notch-toughness. Quenching and tempering, however, brought about a very fine dispersion of carbide particles, and with it, a finer effective ferrite grain size. The improvement in notch-toughness accompanying these changes in microstructure is shown at the bottom of Fig. 4, where the temperature corresponding to an average Charpy value of 15 ft-lb is listed.

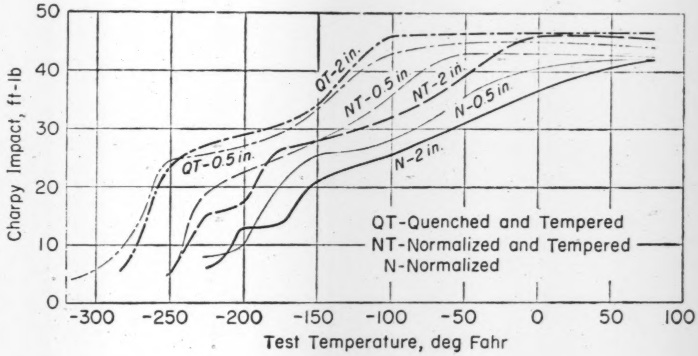
Quenching and tempering may be less effective in the case of lower-hardenability steels, as demonstrated for "2215" plate in Fig. 5(*a*). Here, the curve for quenched and tempered ½-in. plate lies slightly above and to the left of the curve for normalized plate, but not nearly as much as for 2317 in Fig. 3. On the other hand, in higher-hardenability steels quenching and tempering may markedly improve the low-temperature notch-toughness even for fairly heavy sections. For instance, Fig. 5(*b*) shows that, as compared to normalizing, quenching and tempering 2-in. thick 4815 steel plate improved its notch-toughness at low temperature as much as it did the ½-in. thick plate. Quenching and tempering 2810 steel also improved its notch toughness slightly, as compared to the normalized and tempered condition (Fig. 5(*c*)).

Tempering also improved the notch-toughness of normalized 4815 plate, both ½ and 2 in., whereas tempering normalized 2317 and "2215" had little effect. The improvement in the 4815 grade is due to the fact that this steel, as normalized, contained a small amount of martensite.

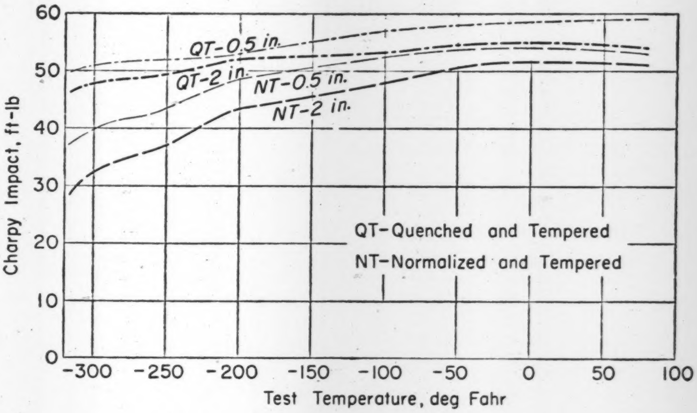
The foregoing data indicate that by proper heat treatment, notch-toughness of suitably alloyed steel plate at low temperature can be improved by a very



(*a*) "2215" steel plate.



(*b*) 4815 steel plate.



(*c*) 2810 steel plate.

FIG. 5.—Effect of Heat Treatment and Plate Thickness on Keyhole-Notch-Toughness of "2215," 4815, and 2810 Steel Plate. Longitudinal Specimens.

substantial amount. In some instances such heat treatment may not be feasible or economical, or it may lead to an undesirable amount of distortion or to other difficulties. When heat treatment is practical, however, it permits the most efficient utilization of the alloying elements in the steel. Of the heat treatments discussed, normalizing will generally result in some improvement over the hot-rolled condition, but quenching and tempering will be even more effective, providing the steel can be hardened properly by quenching.

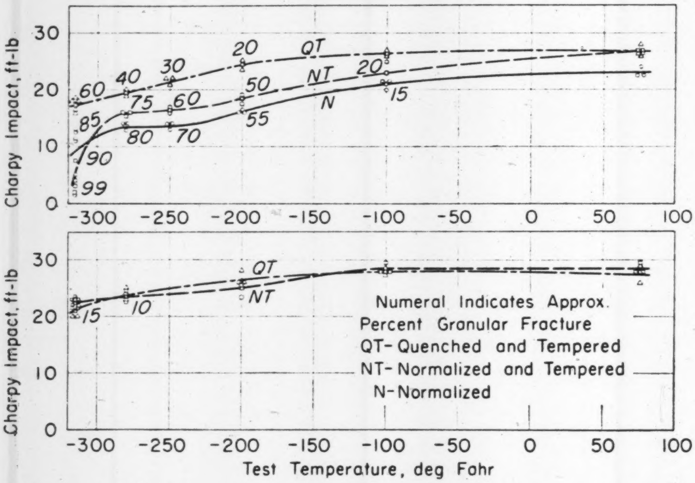


FIG. 6.—Results of Impact Tests for Half-Size Keyhole-Notch Specimens from Tubes of 2317 and 2810 Steels.

Effect of Plate Thickness:

The effect of differences in plate thickness depends both on composition and heat treatment. These factors affect the microstructure of the steel which, in turn, influences the notch-toughness at low temperature.

After hot rolling, thicker plate normally cools more slowly than thinner plate, also the thicker plate usually finishes hot rolling at a higher temperature. These factors tend to produce coarser grains and other structural differences in the thicker plate, which make it less tough at low temperature than thinner plate. Thus, in Fig. 3 the curve for 2-in. thick plate of hot-rolled 2317 steel lies considerably to the right of the corresponding curve for ½-in. plate.

In steels having relatively low hardenability, a similar, though less marked difference may exist in normalized or normalized-and-tempered plates of different thickness, as illustrated for 2317 steel in Fig. 3 and for "2215" in Fig. 5(*a*). With greater hardenability the differences in cooling rate due to differences in plate thickness have less effect on microstructure, and consequently notch-toughness is affected less than in lower-hardenability steels. Thus, the curves for normalized ½-in. plate and for 2-in. plate of 4815 steel in Fig. 5(*b*) lie fairly close together.

In the quenched-and-tempered condition, thickness of the plate will have little or no effect so long as the plate is effectively hardened in the quench. As an example, very similar notch-toughness curves were obtained for ½-in. plate and 2-in. plate of 4815 steel when quenched and tempered (Fig. 5(*b*)) and similarly for quenched-and-tempered 2810 plate (Fig. 5(*c*)). At some larger size, or for steels having less hardenability than these, a definite effect of plate thickness would be expected.

Comparison of Steels:

The data presented here are not intended to show, except in a general way the relative merits of the grades investigated for service at low temperature. To establish within close limits the minimum service temperature for which any one of the grades is suitable, with respect to some selected standard, would necessitate testing samples from several heats of that grade. For these reasons, the Charpy values reported for each steel should be considered only as examples and not necessarily as representative of the grade.

The over-all behavior of the four steels investigated would rate them in the following order with respect to the temperature at which their notch-toughness first reaches a relatively low value:

1. "2215" (highest temperature)
2. 2317 and 4815
3. 2810 (lowest temperature)

The above arrangement is in the order of increasing nickel content, although this is only one of the factors responsible for increased notch-toughness at low temperature.

RESULTS FOR TUBING

In determining the effect of temperature on notch-toughness of specimens from seamless tubing of 2317 and 2810 steels, keyhole-notched Charpy specimens one-half the standard thickness, that is 0.394 in. by 0.197 in. in cross-section, were used. Results for all specimens tested are shown in Fig. 6.

Tempering normalized 2317 tubing improved its notch-toughness slightly at the higher test temperatures but did not lower the temperature at which toughness began to drop rapidly. Quenching and tempering improved the notch-toughness at low temperatures to such an extent that only a slight decrease in Charpy value was observed at the lowest temperature used, −315 F.

For the 2810 steel, practically no difference was found between the normalized-and-tempered tubing and the quenched-and-tempered tubing.

These results for tubing are not directly comparable with those for the corresponding steels in plate form. One reason for this is that although half-thickness specimens absorb less energy at relatively high temperature, they generally can be cooled to a lower temperature than full-thickness specimens before they become relatively brittle (6). Another factor is the difference in response to heat treatment due to differences in thickness of the original material. The tubing was considerably thinner than even the ½-in. plate, and consequently the 2317 tubing hardened more thoroughly when normalized or quenched. As a consequence, this tubing was harder than the plate after tempering, even though the tubing was tempered at a slightly higher temperature. For the 2810 steel, which has considerably greater hardenability than 2317, the difference in hardness between quenched-and-tempered plate and tubing is somewhat less. When normalized, the tubing, because of its thinner section hardened more thoroughly than the plate; consequently for the tubing there was relatively little difference between the normalized-and-tempered condition and the quenched-and-tempered condition.

SUMMARY

This investigation provides data on the notch-toughness of "2215," 2317, 4815, and 2810 alloy steels in the form of ½-in. and 2-in. thick plate, and of 2317 and 2810 steels in the form of thinner-walled tubing, at temperatures from room temperature to −315 F. These results may be summarized briefly as follows:

1. Notch-toughness of all four steels decreased gradually at first as the test temperature was lowered. For the three lower-alloy steels, this gradual decrease was followed by a more abrupt drop at relatively low temperature.

2. No significant difference in notch-toughness was found between specimens from the centerline of the plate and those from a location midway between center and edge.

3. Charpy values were considerably higher for longitudinal specimens than for transverse specimens, except at low temperatures where both types of specimens were relatively brittle. This was probably due to the directionality resulting from straight-away rolling (no cross rolling).

4. Notch-toughness at low temperature was greatly improved by suitable heat treatment. In general, normalized plate was better than hot-rolled plate, and quenching and tempering resulted in further improvement.

5. The effect of plate thickness was found to depend upon hardenability of the steel and upon its heat treatment. Specimens from ½-in. thick plate were found to have greater toughness at low temperature than those from 2-in. thick plate, except when hardenability was such that the thicker plate responded to heat treatment as effectively as the thinner plate.

6. In general, the temperature at which the Charpy value dropped below some selected value, for instance 15 ft-lb, was highest for "2215," next lowest for 2317 and 4815, and lowest for 2810.

7. The effect of decreasing temperature on notch-toughness of half-size Charpy specimens from tubing was less than for full-size Charpy specimens from ½-in. or heavier plate. This difference is due partly to the different size of specimen used, and probably to a lesser extent, to differences in cooling rate for the same nominal heat treatment.

Acknowledgments:

The authors are indebted to many of their associates, particularly at South Works and Gary Works of United States Steel Co and in the National Tube Co, for supplying the material for this investigation heat treated and partially machined to finished size. The tensile data in Table II also were furnished by Gary Works. The assistance of J. B. Carlin and E. M. Patterson in the experimental work is gratefully acknowledged.

REFERENCES

(1) T. N. Armstrong and G. R. Brophy, "Some Properties of Low Carbon 8½ Per Cent Nickel Steel," Presented at Annual Conference of Petroleum Division, Am. Soc. Mechanical Engrs., held at Houston, Texas, Oct. 5–8, 1947.

(2) G. R. Brophy and A. J. Miller, "The Metallography and Heat Treatment of 8 to 10 per cent Nickel Steel," *Transactions*, Am. Soc. Metals, Vol. 41, p. 1185 (1949).

(3) H. D. Newell, J. A. Manfre and M. A. Cordovi, "New 9% Nickel Steel Tubing—Properties and Precessing," *Materials and Methods*, Vol. 25, p. 62 (1947).

(4) H. W. Gillett, "Impact Resistance and Tensile Properties of Metals at Subatmospheric Temperatures," Am. Soc. Testing Mats. (1941). (Issued as separate publication, *STP No. 47*.)

(5) H. W. Gillett and F. T. McGuire, "Report on Behavior of Ferritic Steels at Low Temperatures: Parts I and II" (Report of War Metallurgy Committee of National Research Council), Am. Soc. Testing Mats. (1945). (Issued as separate publication, *STP. No. 63*.)

(6) H. Habart and W. J. Herge, "Sub-size Charpy Relationships at Sub-Zero Temperatures," *Proceedings*, Am. Soc. Testing Mats., Vol. 39, p. 649 (1939).

DISCUSSION

Mr. H. C. **Boardman**[1] (*by letter*).—This paper is intensely interesting to the writer because of its bearing on the ASTM Tentative Specification for Steel Plates For Pressure Vessels For Service at Low Temperatures (A 300 - 50 T) to which, under Scope, the following note is being recommended to be added:

> Note.—It should be recognized that fabricating processes, such as forming and welding, may affect the impact properties of steel so that the impact properties of the plate in a fabricated pressure vessel may differ from the impact properties of the plate as tested.

Without such a cautionary note, the user of pressure vessels made of steel purchased to ASTM Specification A 300 naturally might assume without question that his vessel shells have a Charpy keyhole notch impact value of at least 15 ft-lb at the temperature listed for the material in Specification A 300. In fact, without this cautionary note, code writers might innocently specify that materials ordered to Specification A 300 need not be impact tested, after fabrication, either in plates or welds, provided the lowest service temperature is not below the minimum temperature listed for the material in Specification A 300.

Therefore, the writer's sole purpose in this brief discussion is to urge the authors to conduct a supplementary test program to determine and report the effects on steels purchased to Specification A 300 of cold working, welding, and thermal stress relieving, separately or in combination, so that fabricators and users will be aware of and make allowance for these effects.

Otherwise stated, the users of pressure vessels to be operated at temperatures below −20 F should have data enabling them to appraise what they are getting in the completed vessel for the extra cost of A 300 materials.

Mr. M. A. **Scheil**[2] (*by letter*).—The effect of plate thickness on the low-temperature notch impact strength is of interest especially since the authors find a dependence upon hardenability of the steel and upon its heat treatment. It would be of interest to have data which would illustrate the effect of greater plate thicknesses or slabs which could then be used to interpret the size effect in forgings. Of particular interest to several people would be the results of Charpy impact tests at low temperature on nickel-steel plates between 2 to 6 in. in thickness, as it is believed by several investigators that erratic results begin to appear in plate or billets above 4 in. in thickness. This may not be true of the steel compositions studied by the authors, and therefore it is of general interest.

It is noted that all tempered specimens were quenched after tempering. Supposedly this was done to avoid the possibilities of temper embrittlement. However, we do not believe that this would be possible in a large fabricated structure and particularly where Code requirements for unfired pressure vessels must be met. It is possible that the authors may have some data which would compare the standard thermal stress relief where vessels are furnace cooled to 600 F and may then be permitted cooling in still air.

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[1] Director of Research, Chicago Bridge & Iron Co., Chicago, Ill.

[2] Director of Metallurgical Research, A. O. Smith Corp., Milwaukee, Wis.

Of principal interest is the behavior of the materials investigated after quenching and tempering. The authors are careful to say that when heat treatment is practical it permits the most efficient utilization of the alloying elements in the steel. Would the authors consider quenched-and-tempered plate practical for pressure vessel application? Could they say whether low temperature notch impact strength would be benefited in the zone affected by welding to the same extent that quenched-and-tempered plate is over normalized-and-tempered plate?

The above information is of course very important to the fabricator inasmuch as the notch impact strength in the heat-affected zone has the same requirements placed upon it as the parent metal and weld metal.

MESSRS. W. B. SEENS, W. L. JENSEN, AND O. O. MILLER (*authors' closure*).—The authors welcome Mr. Boardman's comments for we fully agree that the properties described in this paper apply only to the base plate before forming or welding.

In reply to Mr. Scheil's discussion, we have no data on thicknesses greater than 2 in., but would anticipate that with increasing thickness the extent of improvement by quenching and tempering over normalizing would diminish with decreasing hardenability. However it is probable that normalizing would improve the properties of even very thick sections of all these steels if the normalizing temperature were considerably below the finish rolling or forging temperature.

There is some doubt whether temper embrittlement would occur to a significant degree in these steels. We have data on effect of cooling rate for only the 9 per cent nickel steel, and these indicate only slightly lower notch toughness at −315 F after furnace cooling from 1050 F as compared to quenching.

The authors do not feel qualified to comment on the suitability of heat-treated plate for specific applications, such as for pressure vessels, nor do we have any data on the effect of welding on the notch toughness of these steels.

NOTCH-TOUGHNESS OF FULLY HARDENED AND TEMPERED LOW-ALLOY STEELS*

By R. L. Rickett[1] and J. M. Hodge[2]

Synopsis

Notch-toughness of several low-alloy steels quenched entirely to martensite and then tempered was measured by means of Charpy keyhole-notch impact tests. In general, low-carbon steels were found to be tougher than higher-carbon steels when the latter were tempered at some higher temperature to produce the same hardness. This conclusion is based on statistical analysis of data, including some from other sources, representing a large number of impact tests at room temperature, made on steels containing approximately 0.10 per cent to 0.65 per cent carbon.

The tendency for notch-toughness to decrease on tempering at about 600 F, previously reported by others, was confirmed. It was found that the magnitude of this loss in notch-toughness depends upon the temperature of test. As the temperature of test was lowered, specimens tempered at 600 F became brittle sooner (at a higher temperature) than specimens tempered at 450 F or at 700–950 F. Cooling quenched specimens to −320 F before tempering, to transform any retained austenite present, did not eliminate the loss in toughness on tempering at 600 F, although it did change the density of the steel, probably because a small amount of retained austenite transformed at the low temperature. It is concluded, therefore, that the presence of retained austenite probably is not responsible for loss of toughness on tempering at 600 F.

Although results of the notch-impact test cannot be utilized directly in design, they often are a useful indication of relative toughness, particularly under the complex stress conditions frequently encountered in service. The purpose of the present investigation was to determine the effect of composition, hardness, and other variables on the notch-toughness of low-alloy steels completely hardened (quenched entirely to martensite) and then tempered.

The Charpy impact test, with keyhole-notch specimens, was used as a measure of notch-toughness. To cover a wide range of composition and hardness, 25 low-alloy steels were investigated (Table I); most of these were commercial grades but some were special heats. These steels ranged in carbon content from 0.09 per cent to 0.64 per cent, and each contained a sufficient amount of one or more of the common alloying elements so that standard impact specimens could be completely hardened throughout by quenching in oil or brine. The fully hardened[3] specimens were tempered for 1 hr at temperatures in the range 300 to 1150

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* Presented at the Fifty-fourth Annual Meeting of the Society, June 18–22, 1951.

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[2] Research and Technology Division, United States Steel Co., Pittsburgh, Pa.

[3] The microstructure of these steels after quenching consisted almost entirely of martensite. A very small amount of higher-temperature transformation products (pearlite or bainite), and a small amount of retained austenite also was present in some steels.

TABLE I.—STEELS INVESTIGATED.

Grade	Composition, per cent[a]								
	C	Mn	P	S	Si	Ni	Cr	Mo	V
3312	0.11	0.45	0.017	0.009	0.18	3.33	1.52	0.03	...
5 Cr-Mo	0.09	0.31	0.014	0.007	0.27	...	5.32	0.54	...
2320	0.21	0.50	0.006	0.013	0.32	3.61	0.18	0.02	..
3 per cent Cr	0.24	0.65	0.013	0.021	0.26	0.05	3.20	0.03	...
3120	0.16	0.65	0.017	0.023	0.34	1.24	0.68	0.02	...
4620	0.21	0.65	0.019	0.035	0.29	1.79	0.12	0.24	...
4320	0.22	0.51	0.015	0.023	0.23	1.85	0.47	0.26	...
8620	0.23	0.76	0.026	0.022	0.23	0.51	0.51	0.22	...
1330	0.34	1.67	0.020	0.028	0.25	...	...	...	...
2 per cent Cr	0.32	0.45	0.007	0.011	0.28	...	1.97	...	...
8630	0.28	0.82	0.024	0.020	0.32	0.55	0.54	0.19	...
9430	0.26	0.96	0.006	0.018	0.45	0.45	0.40	0.15	...
2 Cr-Mo	0.35	0.44	0.010	0.010	0.24	...	2.01	0.49	...
2330	0.27	0.62	0.010	0.020	0.28	3.40	...	...	...
4130	0.32	0.49	0.019	0.025	0.23	...	1.03	0.20	...
1340	0.44	1.66	0.013	0.030	0.24	...	...	...	...
8440	0.38	1.41	0.020	0.029	0.31	...	...	0.14	...
0.5 Mo	0.39	0.43	0.011	0.007	0.19	...	...	0.52	...
8640	0.38	0.87	0.024	0.018	0.31	0.62	0.61	0.21	...
9440	0.37	0.98	0.027	0.023	0.25	0.47	0.41	0.11	...
3140	0.43	0.79	0.013	0.022	0.32	1.23	0.60	0.06	...
2340	0.40	0.81	0.007	0.016	0.32	3.40	0.06	0.01	...
4140	0.46	0.95	0.017	0.030	0.27	0.07	1.00	0.19	...
6150	0.53	0.81	0.016	0.025	0.22	...	0.92	...	0.16
4063	0.64	0.94	0.011	0.016	0.23	...	...	0.28	...

[a] Check analyses except for 3312, 5 Cr-Mo, and 6150, which are ladle analyses.

TABLE II.—HEAT TREATING DATA.

Grade	Initial Condition[a]	Austenitized	Austenite Grain Size	Quenched In	High Temperature Transformation Product	Hardness As Quenched Rc
3312	Nz-T	1600 F.	9	Brine	None	39
5Cr-Mo	Nz-T	"	7-8	Brine	None	38.5
2320	Nz-T	"	8-9	Brine	Trace	45
3 per cent Cr	Nz-T	"	9	Brine	None	48
3120	Nz-T	"	7 Few 5	Brine	0.1–0.2 per cent	42
4620	Nz-T	"	8	Brine	None	46
4320	Nz-T	"	7–8	Brine	None	44.5
8620	Nz-T	"	7–8 Few 5	Brine	Trace	46.5
1330	Ann.	"	8-9	Brine	0.1–0.2 per cent	56
2 per cent Cr	Ann.	"	5(3–6)	Oil	None	49
8630	Ann.	"	6-7 Few 5	Brine	None	50.5
9430	Ann.	"	7–8	Brine	Trace	50.5
2 Cr-Mo	Nz	"	6-7 (5–8)	Oil	None	54
2330	Ann.	"	8	Brine	Trace	51.5
4130	Nz	"	7–8	Brine	None	54
1340	Ann.	"	8 (7–9)	Oil	0.2–0.4 per cent	56
8440	Ann.	"	8	Oil	Trace	55.5
0.5 Mo	Ann.	"	6–7	Brine	Trace	55.5
8640	Ann.	"	8–9	Oil	None	56.5
9440	Ann.	"	6–8	Oil	0.2–0.3 per cent	53
3140	Ann.	"	9	Oil	0.1 per cent	56
2340	Nz-T	"	8–9	Oil	Trace	56.5
4140	Nz-T	"	7–8	Oil	None	58.5
6150	Nz-T	"	7–8	Oil	None	...
4063	Nz-T	"	6	Oil	None	...

[a] Condition prior to austenitizing for final heat treatment

Nz: normalized

Nz-T: normalized, tempered

Ann: "full anneal" at 1500 F.

F, then cooled in air. Results for these steels, tested at room temperature, were supplemented by comparable data from other investigations by the authors and their associates and by published data from another source.

As is well known, the notch-toughness of steel depends on the temperature of test as well as on other factors. Although it was not feasible to repeat the entire testing program at several temperatures (approximately 450 specimens were tested at room temperature), suitably tempered specimens of several of the steels were tested over a range of temperature.

To explore the possibility that in some steels retained austenite might affect the notch-toughness, quenched specimens were cooled to −320 F, before they were tempered, to transform retained austenite that might be present. Notch-toughness and also the density of these quenched, refrigerated, and tempered specimens were compared with the toughness and density of quenched-and-tempered specimens.

MATERIAL AND PROCEDURE

The steels investigated, Table I, include a number of SAE or AISI low-alloy steels and a few non-standard alloy steels in the 0.10 to 0.65 per cent carbon range.

Standard Charpy keyhole-notch impact specimens were austenitized at 1600 F, preliminary tests having indicated that in all the steels all carbides were substantially dissolved at this temperature. It was necessary first to normalize some of the steels at 1700 F in order to accomplish this. The condition of each steel prior to austenitizing is listed in Table II. The specimens, packed in cast iron chips in a closed container, were heated for 3 hr, of which slightly over 2 hr was required to reach 1600 F. This method of protection from scaling and decarburization was selected after preliminary tests indicated it to be the best of those readily available. Some decarburization was noted around the notch but at most was only partial and very shallow (less than 0.001 in.). The austenite grain size produced under the foregoing conditions is given in Table II. After austenitizing, the specimens were quenched in oil at 110 to 130 F or in brine at room temperature, as noted in Table II; the resulting microstructure was essentially completely martensitic in all instances. Specimens were tempered in molten salt, for 1 hr unless otherwise noted, after which they were cooled in air; tempering was started within 30 min after quenching, but not before the specimens had cooled to room temperature.

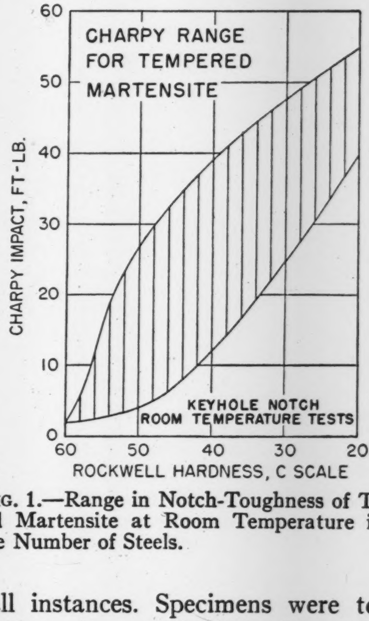

FIG. 1.—Range in Notch-Toughness of Tempered Martensite at Room Temperature in a Large Number of Steels.

Before tempering, some quenched specimens were refrigerated by immersion in liquid nitrogen (−320 F) for 1 min, which was just a few seconds longer than the time required for the center of the specimen to reach the temperature of the bath. The elapsed time between quenching and refrigeration was always less than 1 min. The specimens were then tempered, unless they were to be tested in the "as quenched and refrigerated" condition.

NOTCH-TOUGHNESS AT ROOM TEMPERATURE

It was found that the impact properties of tempered martensite, as represented by the steels studied in this investigation, covered a fairly wide range and that this spread in properties was particularly marked in the hardness region between 35 and 50 Rockwell C. The spread in impact properties among the steels studied in this investigation is illustrated by Fig. 1. This spread is contrary to the general observation that the ordinary mechanical properties of tempered martensite, within certain limits of hardness or tensile strength, are relatively constant regardless of the type of steel (**1**, **2**).[4] It is not, however, entirely contradictory, since the widest spread here is in the hardness region beyond that to which the foregoing generalization applies and since notch-toughness is, in general, more sensitive to metallurgical change than are tensile properties. Nevertheless, it is desirable to analyze these results in more detail and to attempt to arrive at the factors involved in this wide range of impact values for a given hardness. Among the factors that may affect these impact values are carbon content and tempering temperature. All of the data were therefore analyzed on the basis of these two variables in order to evaluate their effect.

Effect of Carbon:

The steels were separated into five groups according to carbon content, and for each group individual room temperature Charpy values were plotted as a function of hardness, with the results shown in Fig. 2. These data include not only those from the present investigation but also the results of other measurements of notch-toughness of tempered martensite at room temperature made by the authors and their associates and by Gillett and McGuire (**3**). Standard statistical methods were used to calculate the "average" relation between hardness and notch-toughness for each of these groups, and also the standard deviation for each. These average (regression) curves and the 2 σ limits (twice the standard deviation) are shown in Fig. 2. For these calculations, the relation between hardness and Charpy value was assumed to be linear. This assumed relation is believed to be sufficiently accurate for comparative purposes, when considering the large volume of data represented here, although it is known not to be true for any one of the steels considered individually.

Average curves representing the five groups of data in Fig. 2 are shown together for comparison in Fig. 3. Curves for the two lower carbon ranges nearly coincide, and hence only four are shown in Fig. 3. The dashed lines in Fig. 3 represent the 95 per cent confidence intervals for the respective average, or regression, curves. It is evident from these curves and their respective confidence limits that over most of the hardness range represented, carbon content of the steel has a real, and in many cases a very marked, effect on notch toughness at any selected hardness. For comparable hardness, and hence for comparable tensile strength, lower-carbon steels are tougher than higher-carbon steels. This conclusion is, of course, valid in only a general sense; comparison of individual pairs of steels might in some instances show the opposite relationship. Also, it may be strictly applicable only to steels heat treated as these were, that is, quenched entirely to martensite and then tempered. Nevertheless, the conclusion is a very important one and indicates that for applications in which maximum notch-toughness is desirable, low-carbon steels are superior to high-carbon steels, so long as they can be hardened throughout and the desired hardness and strength can be obtained after tempering

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[4] The boldface numbers in parentheses refer to the list of references appended to this paper see p. 943.

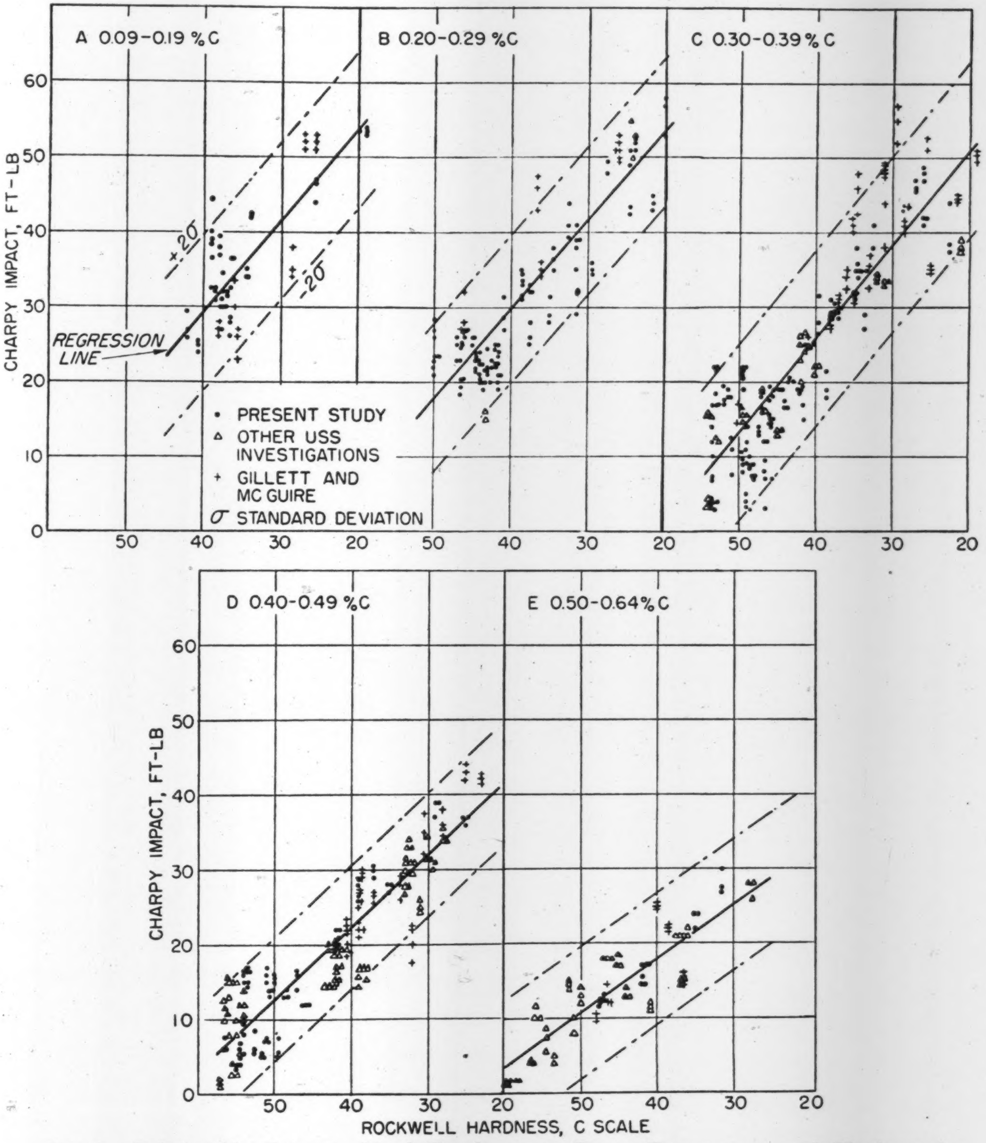


FIG. 2.—Relationship Between Notch-Toughness at Room Temperature and Hardness for Tempered Martensite in Each of Five Ranges of Carbon Content. Charpy Keyhole-Notch Specimens.

The same data were analyzed by the method of multiple regression, leading to the following equation:

$$\text{Charpy Value (ft lb)} = 79.0 - 1.08$$
$$\text{(Rockwell C hardness)} - 29 \text{ (per cent carbon)}$$

Precision of this equation was calculated to be:

$$\sigma^2(Y_1) = 0.106\,[1 + 0.0126\,(x_1 - 40.6)^2 + 75.3\,(x_2 - 0.36)^2]$$

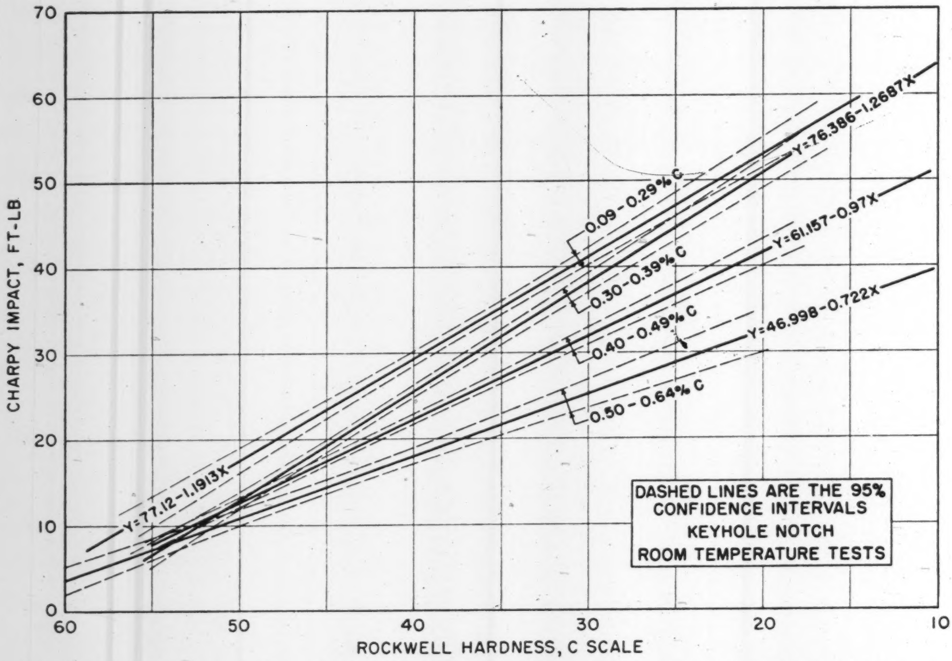


FIG. 3.—Comparison of Curves From Fig. 2 Showing the Effect of Carbon on the Relationship Between Hardness and Notch Toughness of Tempered Martensite at Room Temperature.

where:

$\sigma^2(Y_1)$ = mean-square deviation of the predicted Charpy value,

x_1 = Rockwell hardness and

x_2 = Carbon content.

In deriving this equation, the relationships between hardness, Charpy value, and carbon content were assumed to be linear. Although the equation has the advantage over the analysis represented in Fig. 3 that it does not require dividing the data up into arbitrary carbon ranges, both methods were found to account for the same proportion, about 85 per cent, of the total variability of the data. The factors responsible for most of the remaining variability have not been determined; differences among triplicate specimens account for only a small portion of it (about 1 per cent).

Effect of Tempering Temperature:

Another way of looking at these same data is on the basis of hardness as tempered at a particular temperature, since this will vary with carbon content and to some extent with changes in other elements in the steel. The data are grouped according to tempering temperature in Fig. 4. The range of hardness within each group results largely from variations in carbon and other elements in the steels. Results of individual tests are plotted, as in Fig. 2, and the data are from the same sources. Whereas in Fig. 2 a straight line may be considered to represent each group of data sufficiently well for comparative purposes, this is true in Fig. 4 for only the 860 to 1050 F and 1060 to 1250 F groups. The regression line was calculated for each of these, first for the data obtained in the present investigation alone, then for all of the points represented in the chart, with very good agreement between the two. For the lower ranges of tempering temperature the data obviously depart from a linear relationship over at least part of the range covered. For these, the regression line based on an assumed linear relationship would have no meaning; therefore

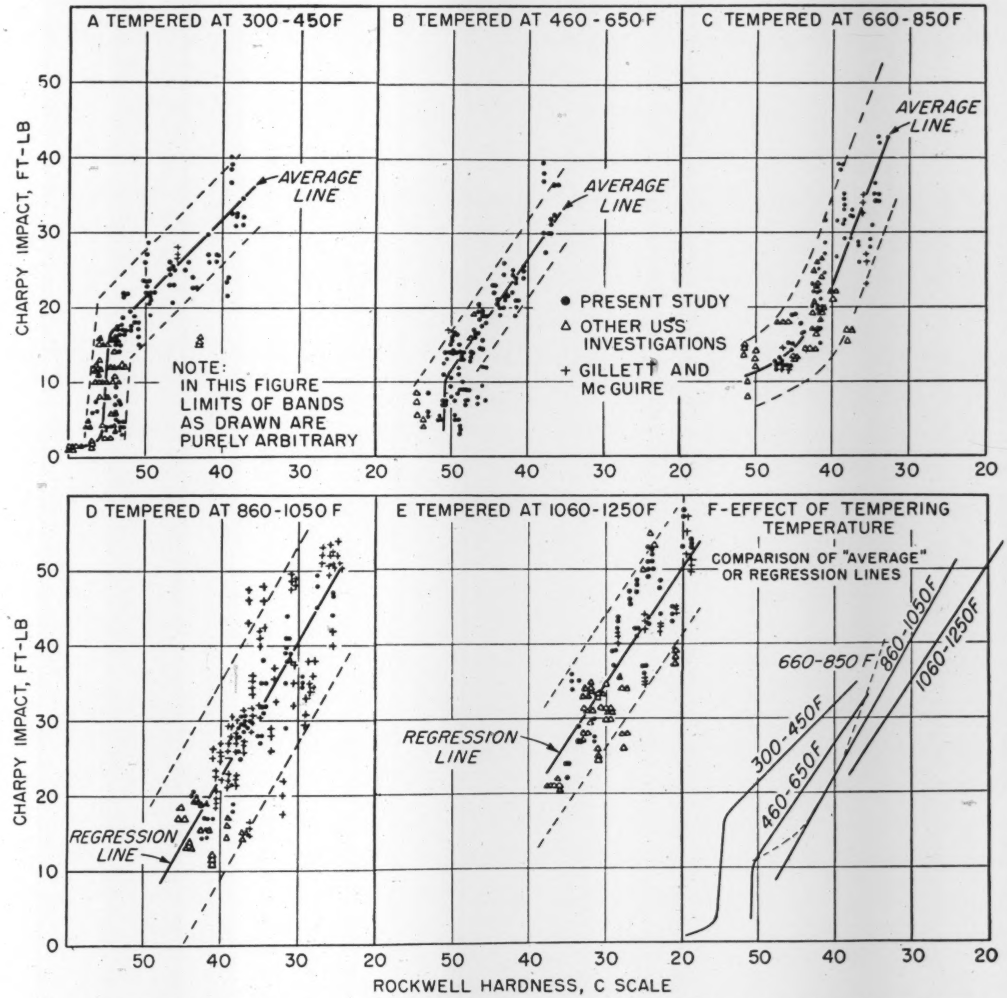


Fig. 4.—Relationship Between Notch-Toughness and Hardness of Tempered Martensite at Room Temperature for Each of Several Ranges of Tempering Temperature. Same Data as Figs. 1, 2, and 3.

an "average" curve determined by inspection was drawn to represent the data. Location of such a line is of course largely a matter of personal judgment. Standard deviation was not calculated, but bands are drawn in each chart of Fig. 4 to include most of the points. The "average" or regression lines are shown together for comparison in Fig. 4 (*F*).

Figure 4(*A*) indicates that as tempered in the range 300 to 450 F, notch-toughness of those steels harder than about 57 Rockwell C is very low. Steels in the hardness range 53 to 57 Rockwell C vary considerably in notch-toughness; sometimes a wide variation was observed even among specimens of a single steel. Those steels softer than 53 Rockwell C when tempered in this temperature range have higher and more consistent notch-impact values The curve in Fig 4(*A*) is quite similar in form to the curve relating notch-impact to *test temperature* for many steels which undergo a transition from relatively high to much lower notch-toughness over a fairly narrow test-temperature range; in Fig. 4(*A*) the "transition" is over a range of hardness rather than temperature of test.

When tempered at 460 to 650 F, Fig. 4(*B*), the entire curve is lower and the "transition" behavior, evidenced by relatively wide scatter, seems to be moved to lower hardness. The line representing tempering at 660 to 850 F, Fig. 4(*C*), appears to curve at the high-hardness end in the reverse direction to the trend in Figs. 4(*A*) and 4(*B*); additional data in this region would be required to determine whether the indicated behavior is real. When tempered at 860 to 1050 F, Fig. 4(*D*) or at 1060 to 1250 F, Fig. 4(*E*), the steels included are all softer than about 45 Rockwell C, and there is no evidence that the relationship between notch-toughness and hardness is other than linear.

Comparison of all the curves, Fig. 4(*F*), shows a definite trend toward lower notch-toughness for comparable hardness the higher the tempering temperature. It is significant that this lower toughness is not confined solely to the tempering range 460 to 650 F in which notch-toughness is often observed to remain constant or decrease as tempering temperature increases, but continues over the entire range investigated. The practical conclusion from either Fig. 3 or Fig. 4(*F*) is the same: highest notch-toughness of tempered martensite is obtained by using a lower-carbon steel tempered at a relatively low temperature rather than a higher-carbon steel which must be tempered at a higher temperature to bring it to the same hardness.

Effect of Refrigeration Before Tempering:

Retained austenite present in a steel as quenched might be expected to affect its notch-toughness when tempered in the temperature range where the retained austenite would either transform or be "conditioned" so that it would transform to martensite when cooled. Since transformation of austenite is accompanied by a decrease in density (increase in volume) whereas martensite on tempering undergoes an increase in density (decrease in volume), density measurements were made on a few steels as quenched and after tempering as an indication of the possible presence of retained austenite. Other specimens were cooled in liquid nitrogen to about −320 F immediately after quenching, since Fletcher and Cohen (4) have shown that such treatment results in substantially complete transformation of retained austenite. After subsequent tempering, impact tests and density measurements were made. Results of these measurements and tests are shown in Fig. 5.

The upper portion of Fig. 5(*A*) shows the variation of notch-impact with tempering temperature for three steels, and the lower portion the corresponding density curves. Looking first at the curves representing effect of tempering on Charpy value, top of Fig. 5, it is seen that all of them are quite similar in shape. Up to about 400 F, tempering raises the notch-impact value; between 400 F and about 600 F the curves remain nearly horizontal or drop; on tempering above 600 F the notch-impact value again increases. This behavior, which was exhibited by practically all the steels investigated, is well known and is not confined to the Charpy keyhole-notch impact test (5, 6, 9).

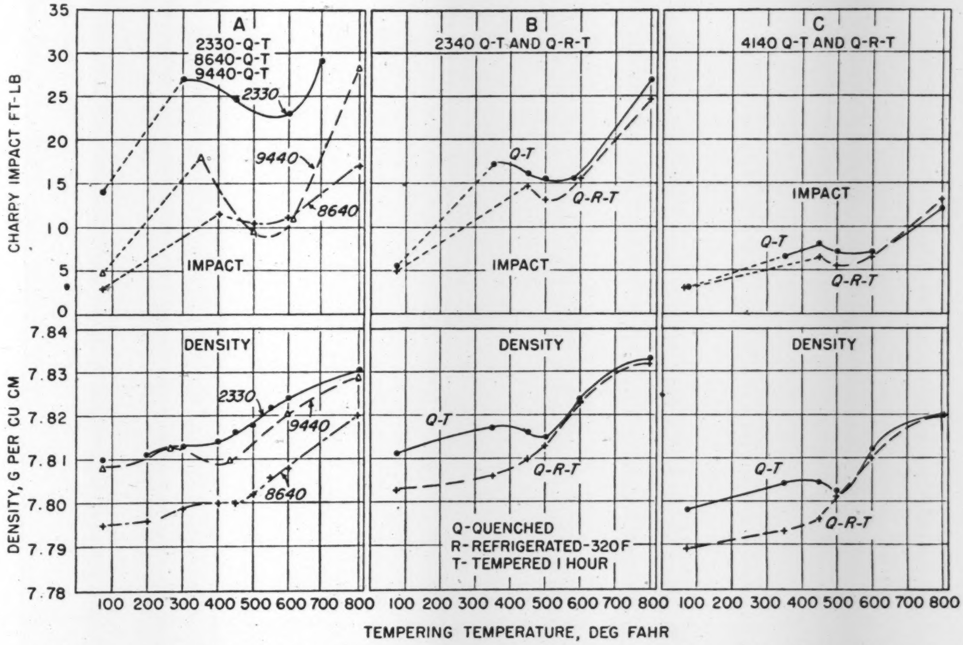
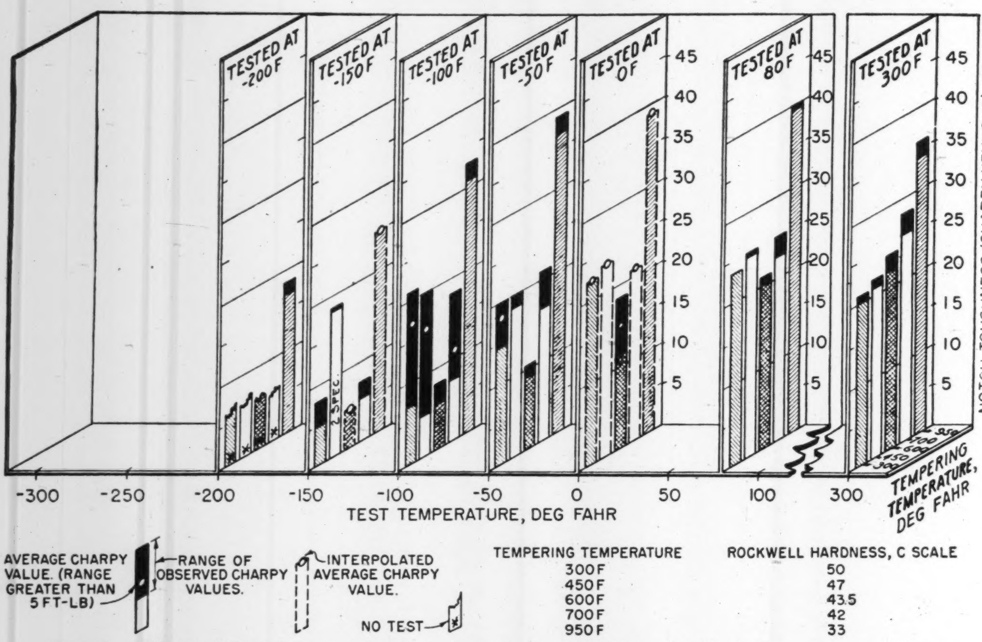


FIG. 5.—Effect of Tempering and of Refrigeration Before Tempering on Notch-Toughness and Density of Selected Low-Alloy Steels.

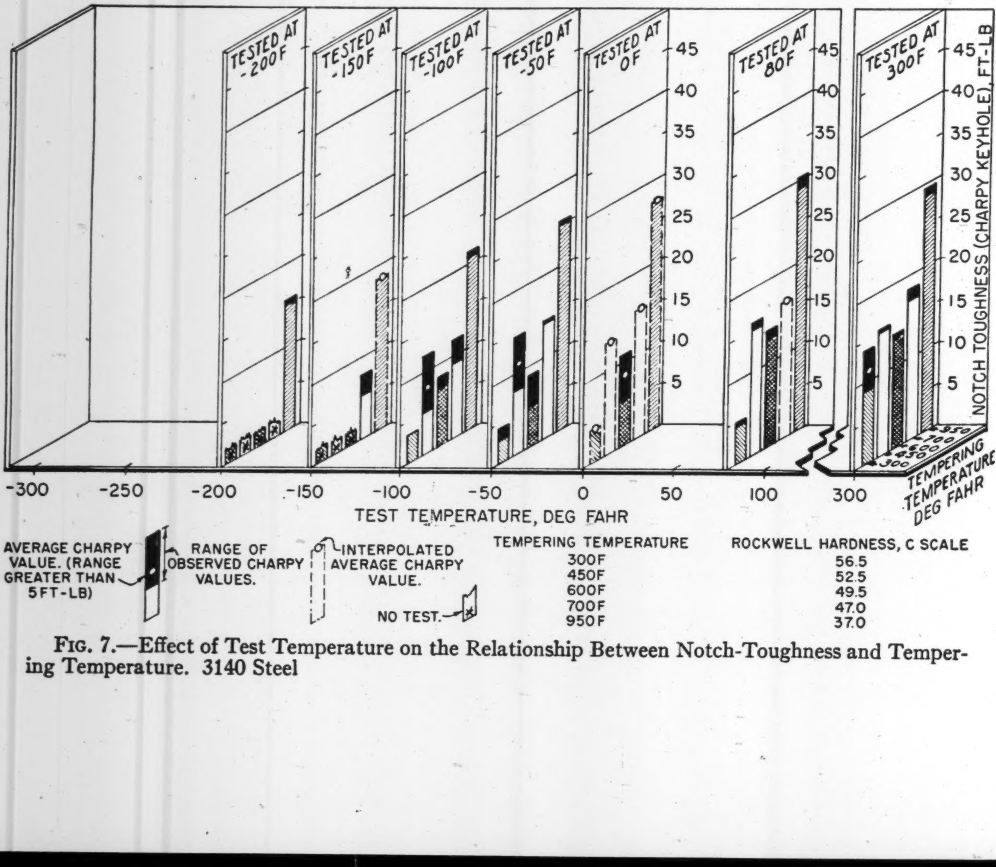
Density, like notch-toughness, does not rise continuously on tempering but tends to level off, or even decrease, on tempering in an intermediate temperature range, which for density is in the neighborhood of 400 F. This behavior is in accord with the supposition, but does not prove conclusively, that a small amount of austenite is present in the steel as quenched and that this austenite transforms on tempering at 350 to 520 F, as has been shown by Antia, Fletcher and Cohen (7).

For specimens refrigerated to −320 F after quenching, then tempered, Figs. 5(*B*) and 5(*C*), the notch-impact behavior is substantially unchanged except possibly for a slight general lowering of the as-tempered values. The density curve, however, is quite different; density after refrigeration is lower, and on tempering it increases continuously. This behavior is strong evidence that some austenite is present in the steel as quenched. Impact behavior apparently is little affected by the presence of this



TEMPERING TEMPERATURE	ROCKWELL HARDNESS, C SCALE
300F	50
450F	47
600F	43.5
700F	42
950F	33

FIG. 6.—Effect of Test Temperature on the Relationship Between Notch-Toughness and Tempering Temperature. 8630 Steel



TEMPERING TEMPERATURE	ROCKWELL HARDNESS, C SCALE
300F	56.5
450F	52.5
600F	49.5
700F	47.0
950F	37.0

FIG. 7.—Effect of Test Temperature on the Relationship Between Notch-Toughness and Tempering Temperature. 3140 Steel

austenite. Grossmann (8) reports this same behavior, with very few exceptions, for ten steels in addition to the data for 2340 and 4140 included in Fig. 5 Incidentally, as shown for SAE-AISI 2330 and 2340 in Fig. 5, tempering at about 600 F was found to decrease the notch-toughness of nickel-alloy steels as well as others, contrary to a published statement (6) that nickel steels do not exhibit this behavior.

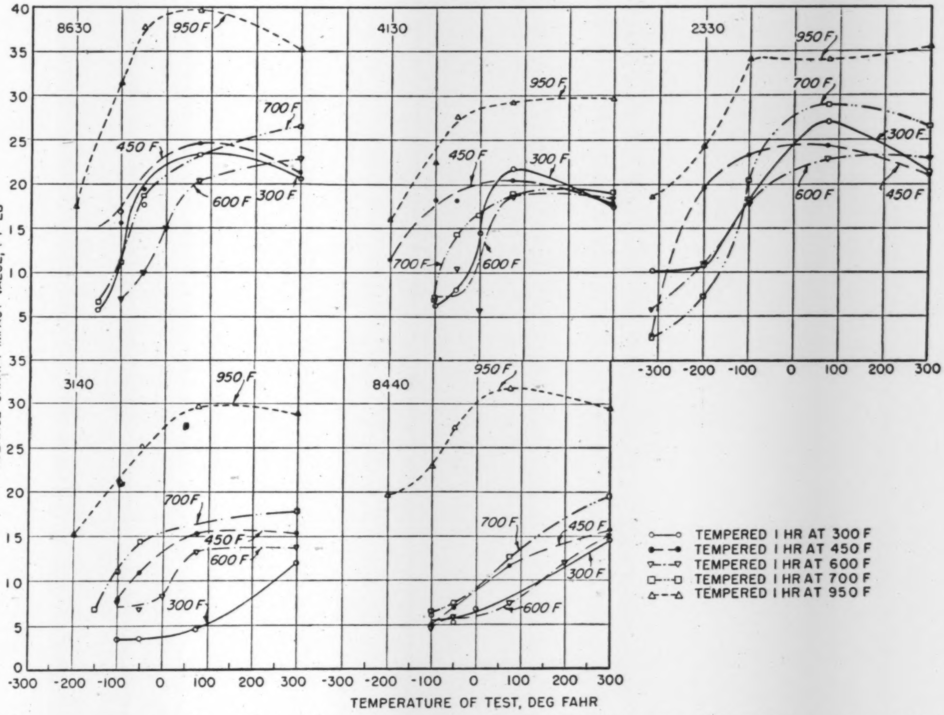


FIG. 8.—Effect of Test Temperature on Average Charpy Value for Five Steels Tempered at 300–950 F.

EFFECT OF TEMPERATURE ON NOTCH-TOUGHNESS

It is well known that the notch-toughness of ferritic steels decreases at low temperatures. Tests were made to determine whether the shape of the characteristic curve relating notch-toughness at room temperature to tempering temperature (as shown in the upper part of Fig. 5(*A*)) is altered if some other testing temperature is used. Five steels selected from the group previously investigated were tested at various temperatures within the range −315 to +300 F, after being quenched and tempered at 300, 450, 600, 700, or 950 F.

The results, illustrated for two of the steels in Figs. 6 and 7, show that the magnitude of the dip in the curve relating notch-toughness to tempering temperature is definitely influenced by the test temperature. When tested at 300 F, toughness rises continuously or exhibits only a shelf or slight dip corresponding to tempering at approximately 600 F. For the five steels tested, the magnitude of this dip increases considerably as the test temperature decreases, although it tends to disappear at very low test temperatures at which most of the specimens are brittle.

Average values for each steel are plotted against test temperature in Fig. 8. In general, as the test temperature was decreased below 300 F, notch-toughness increased slightly at first then decreased gradually. The results show that at most test temperatures the impact values for specimens tempered at 600 F are lower than for specimens tempered at a slightly lower temperature (450 F) or at higher temperatures. In general, this difference is most pronounced in tests at temperatures below room temperature. Specimens tempered at the lowest temperature, 300 F, were the first to reach a low toughness level and those tempered at the highest temperature, 950 F, were the last to reach such a level as test temperature was lowered.

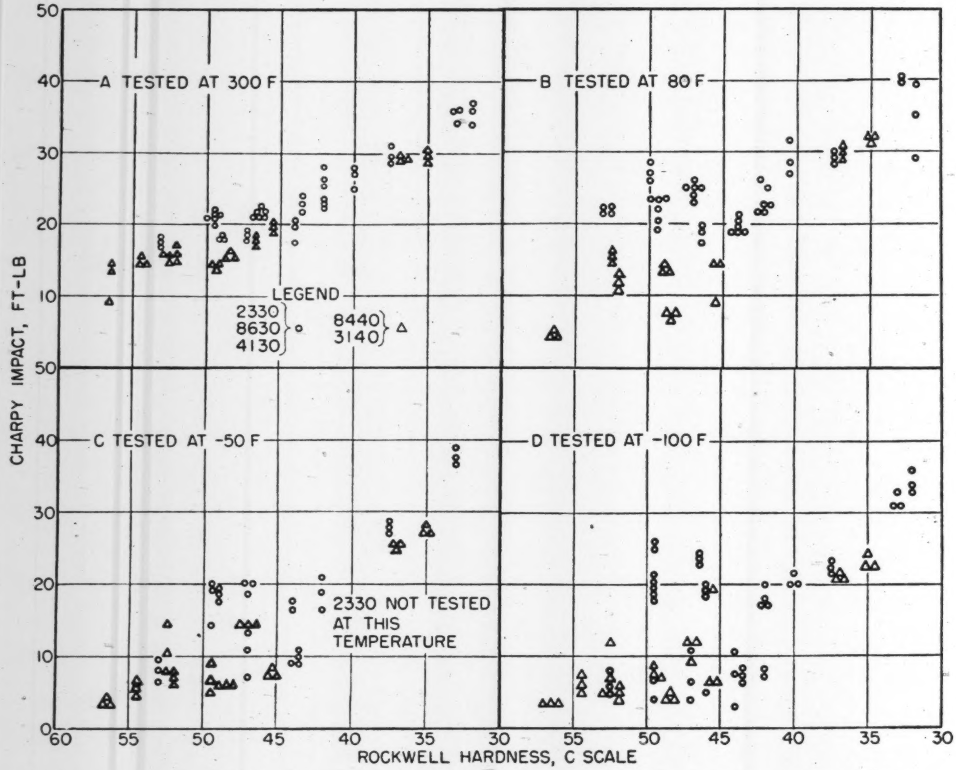


FIG. 9.—Comparison of Relationship Between Hardness and Notch-Toughness for Steels Containing Nominally 0.3 per cent C and 0.4 per cent C at Four Test Temperatures.

As shown in Fig. 9(*A*), notch-toughness of these five steels when tested at 300 F is roughly proportional to hardness, with relatively little spread. When tested at room temperature or below, however, the spread at high hardness (40 Rockwell C or above) increases considerably, the higher carbon steels being the first to drop off in toughness (Figs. 9(*B*), (*C*), and (*D*)). Too few data are available to determine whether the effect of carbon on notch-toughness is as great at other temperatures as it is at room temperature. In general, however, as shown in

Fig. 9, 0.30 per cent carbon steels are tougher at all temperatures tested than 0.40 carbon steels having the same hardness.

SUMMARY

1. On the average, notch-toughness of lower-carbon steels is superior to that of higher-carbon steels when both are quenched entirely to martensite (fully hardened) and tempered to the same hardness, notch-toughness being measured by Charpy keyhole-notch impact tests at room temperature. Average Charpy value for such steels as a class may be computed from the equation:

Charpy value (ft lb) = 79.0 − 1.08

(Rockwell C hardness) − 29 (per cent carbon)

2. The same conclusion may be expressed in terms of the tempering temperature required to produce the desired hardness: tempering at a relatively low temperature (lower-carbon steels) is superior to tempering at the higher temperature required to soften higher-carbon steels to the same extent.

3. In room-temperature tests, notch-toughness was lower after tempering at 600 F than after tempering at a somewhat lower or a higher temperature, as has been shown by others. Refrigeration at −320 F before tempering apparently transformed retained austenite present after quenching, as indicated by changes in density, but did not affect notch-toughness after subsequent tempering. This evidence indicates that relatively low notch-toughness after tempering at 600 F is not caused by retained austenite.

4. When tested at lower temperatures, the effect of tempering temperature on notch-toughness was even greater in many instances than in room-temperature tests. In general, notch-toughness after tempering at 600 F was lower than after tempering at 450 F or at 700 F at all test temperatures except the highest and lowest ones investigated.

5. The relatively small number of tests made over a wide temperature range indicate that steels containing 0.3 per cent carbon are tougher at all temperatures tested than steels containing 0.4 per cent carbon tempered to the same hardness.

Acknowledgment:

The authors gratefully acknowledge the assistance of many of their associates during the course of this work. Much of the experimental work was done by J. M. Ruckman, J. W. Stewart, F. C. Kristufek, and L. A. Luini. The Charpy tests were made by W. B. Seens, W. L. Jensen, and T. F. Berry. We are greatly indebted to L. S. Darken and C. H. Toensing for the highly precise density measurements that were required. Much of the statistical analysis was done by J. C. Butler under the direction of Cuthbert Daniel.

REFERENCES

(1) E. Janitzky and M. Baeyertz, *Handbook* Am. Soc. Metals (1939 edition) pp. 515–518.

(2) W. G. Patton, "Mechanical Properties of NE, SAE and other Hardened Steels," *Metal Progress*, Vol. 43, May, 1943, pp. 726–733.

(3) H. W. Gillett and F. T. McGuire, "Report on the Behavior of Ferritic Steels at Low Temperatures," Parts I and II, Am. Soc. Testing Mats. (1945). (Issued as separate publication *STP No. 63*).

(4) S. G. Fletcher and M. Cohen, "Dimensional Stability of Steel," *Transactions*, Am. Soc. Metals, Vol. 34, pp. 216–237 (1945).

(5) E. C. Bain, "The Alloying Elements in Steel," Am. Soc. Metals (1939).

(6) H. Schrader, H. J. Wiester and H. Siepmann, "Embrittlement of Hardened Steel by Tempering at 250 to 400 C," *Archiv für das Eisenhüttenwesen*, Vol. 21, pp. 21–27 (1950). Abstract-translation by W. M. Baldwin, *Iron Age*, September 14, 1950.

(7) D. P. Antia, S. G. Fletcher and M. Cohen, "Structural Changes During the Tempering of High Carbon Steel," *Transactions*, Am. Soc. Metals, Vol. 32, pp. 290–324 (1944).

(8) M. A. Grossmann, "Toughness and Fracture of Hardened Steel," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 167, pp. 39–79 (1946).

(9) E. J. Ripling and W. M. Baldwin, Jr., "Rheotropic Embrittlement of Steel," *Transactions*, Am. Soc. Metals, Vol. 43, pp. 778–805 (1951).

CREEP PROPERTIES OF TWO TEMPERS OF 63S EXTRUDED ALUMINUM ALLOY*

By O. D. Sherby[1] and J. E. Dorn[1]

Synopsis

The creep and stress-rupture characteristics of 63S extruded aluminum alloy in the T5 and T6 tempers (ASTM alloys GS10A-T5 and GS10A-T6, respectively)[2] were obtained at 90, 212, 300, and 400 F up to 1000 hr rupture time.

Within the range of secondary creep rates tested, the creep properties of 63S-T6 (GS10A-T6) which was air quenched from the extrusion temperature and then artificially aged were superior to those of 63S-T5 (GS10A-T5) over all conditions of test that were investigated. At 300 and 400 F, however, the creep resistances of the two materials approached each other at the lower creep rates.

This report is based on part of an extensive investigation of the creep and stress-rupture properties of aluminum alloys. Data have already been presented on the creep and stress-rupture characteristics of some sheet materials in the precipitation-hardened state (**1**, **3**),[3] some sheet materials in annealed and cold-rolled conditions (**2**, **3**, **4**), and some cast and forged alloys (**5**, **6**). Additional data on the creep properties of aluminum alloys have been presented by other investigators (**7**, **8**). The creep properties of 63S extruded aluminum alloy in the T5 and T6 tempers (ASTM alloys GS10A-T5 and GS10A-T6, respectively) will be described in this report.

The following data were obtained for the two materials, which were investigated at 90, 212, 300, and 400 F:

1. Creep strain as a function of time for various appropriate stresses.
2. Stress *versus* the time to rupture and stress *versus* the secondary creep rate.
3. Stress *versus* the time elapsed for achieving various designated creep strains.

Materials

Different extrusion charges from the same original heat of the 63S alloy were used to produce the two materials for test. Both materials were extruded into rectangular sections 0.062 in. thick and 1.000 in. wide in accordance with standard commercial production procedures. Whereas the 63S-T5 material was aged for 1 to 2 hr at 450 F. after extrusion, the 63S-T6 material was cooled by means of an air blast as it left the extrusion die, after which it was aged for 6 to 8 hr at 350 F.

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* Presented at the Fifty-fourth Annual Meeting of the Society, June 18-22, 1951.

[1] Research Engineer and Professor of Physical Metallurgy, respectively, University of California, Berkeley, Calif.

[2] Tentative Specification for Aluminum and Aluminum-Alloy Extruded Bars, Rods, and Shapes (B 221 - 49 T), and Tentative Specifications for Aluminum-Alloy Extruded Tubes (B 235 - 49 T), 1949 Book of ASTM Standards, Part 2, pp. 553 and 563.

[3] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 953.

The chemical composition, mechanical properties, and grain size for the two test materials are tabulated in Tables I, II, and III, respectively. The data recorded in Table I show that the chemical compositions of the two materials were within the specified tolerance limits. As shown in Table II, the mechanical properties of the materials not only fell within the specified tolerance bands but actually approximated the typical values for these materials. Table III reveals that the two materials had almost equally fine grains; in both materials the grains were slightly elongated in the direction of extrusion. The microstructures of longitudinal and transverse sections of both materials were observed to be typical and representative of commercially extruded 63S alloy in the two respective tempers.

TABLE I.—CHEMICAL COMPOSITION.

ASTM Alloy	Alcoa Alloy and Temper	Composition	Composition, per cent					Other Elements	
			Silicon	Iron	Copper	Manganese	Magnesium	Each	Total
GS10A-T5......	63S-T5	Actual[a]	0.34	0.17	0 2	0.01	0.69	...	...
GS10A-T6......	63S-T6	Actual[a]	0.36	0.19	0.02	0.01	0.69	...	...
		Specified[b]	0.2 to 0.6	<0.35	<0.10	<0.10	0.45 to 0.85	<0.05	<0.15

TABLE II.—MECHANICAL PROPERTIES.

ASTM Alloy	Alcoa Alloy and Temper	Properties	Tensile Strength, psi	Tensile Yield Strength, 0.2 per cent offset, psi	Elongation in 2 in., per cent	Compressive Yield Strength, 0.2 per cent offset, psi
GS10A-T5........	63S-T5	Actual[a]	29 400	24 900	9.5	25 700
		Specified[c]	22 000	16 000	8.0	...
		Typical[d]	30 000	25 000	12.0	...
GS10A-T6........	63S-T6	Actual[a]	39 400	36 000	10.5	36 500
		Specified[c]	32 000	25 000	8.0	...
		Typical[d]	35 000	30 000	12.0	...

TABLE III.—GRAIN SIZE.

ASTM Alloy	Alcoa Alloy and Temper	Grains per mm[a]		
		Thickness	Width	Length
GS10A-T5........	63S-T5	18	16	25
GS10A-T6........	63S-T6	20	19	26

[a] Spectrochemical analysis, mechanical properties, and grain size data were furnished by the Aluminum Company of America.

[b] Specified values were taken from ASTM Specification B 221 - 49 T.[2]

[c] Specified values were taken from ASTM Specification B 235 - 49 T.[2]

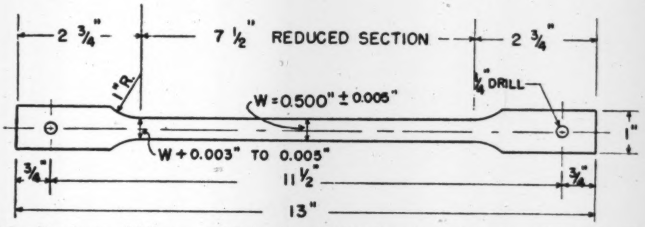
[d] Typical values were taken from "Alcoa Aluminum and Its Alloys," 1947 Edition.

EXPERIMENTAL TECHNIQUE

Detailed descriptions of the creep testing equipment and the method of testing have been recorded in previous publications (1, 3). Sheet-type specimens having the design shown in Fig. 1 were machined with the axis of the specimens parallel to the direction of extrusion. All specimens for test were carefully prepared by the Aluminum Company of America. Rack-and-pinion type extensometers were used; they permitted an evaluation of strains to within ±0.00008. In order to allow the specimen to attain the test temperature, each specimen was mounted in the furnace for approximately 1 hr before the load was applied.

EXPERIMENTAL RESULTS

Strain-*versus*-time curves were plotted on cartesian coordinates for each creep test. Examples of various types of creep curves are shown in Fig. 2. Fig. 2(*a*) illustrates a typical creep curve which depicts the three stages of creep. After a short primary or first stage of creep, giving a monotonically decreasing creep rate, an equally short secondary stage is obtained, exhibiting the secondary or constant creep rate. This is followed by a comparatively longer third stage of creep where the creep rate is constantly increasing until rupture occurs; the point of rupture is indicated by the black dot at the end of the curve. At lower stresses or at lower temperatures each of the three stages is extended over a longer time interval as depicted in Fig. 2(*b*).



Gage length—6 in. Specimen thickness—0.062 in. (nominal). Direction of stress—with grain. Taper—gradual taper from ends of reduced section to middle.

FIG. 1.—Stress-Rupture and Creep Specimen.

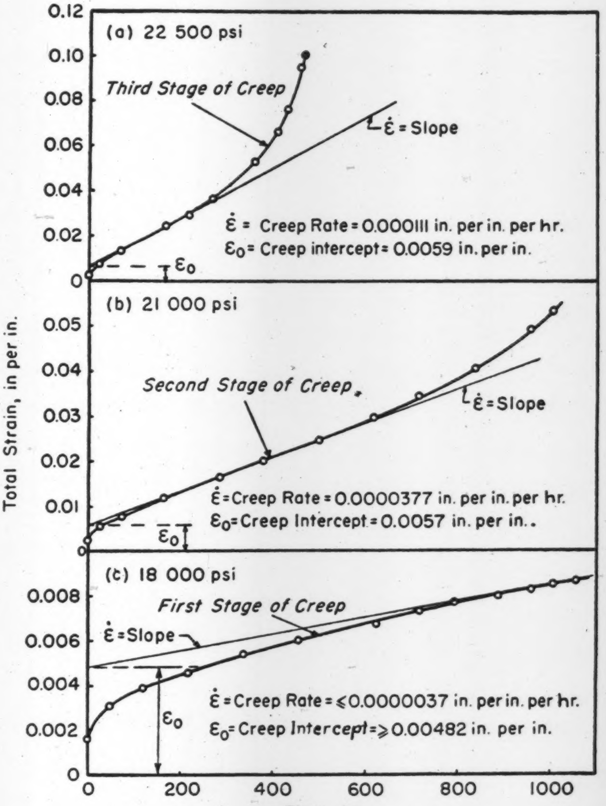


FIG. 2.—Creep Curves of 63S-T5 (ASTM Alloy GS10A-T5) at 212 F Typifying the Three Stages of Creep.

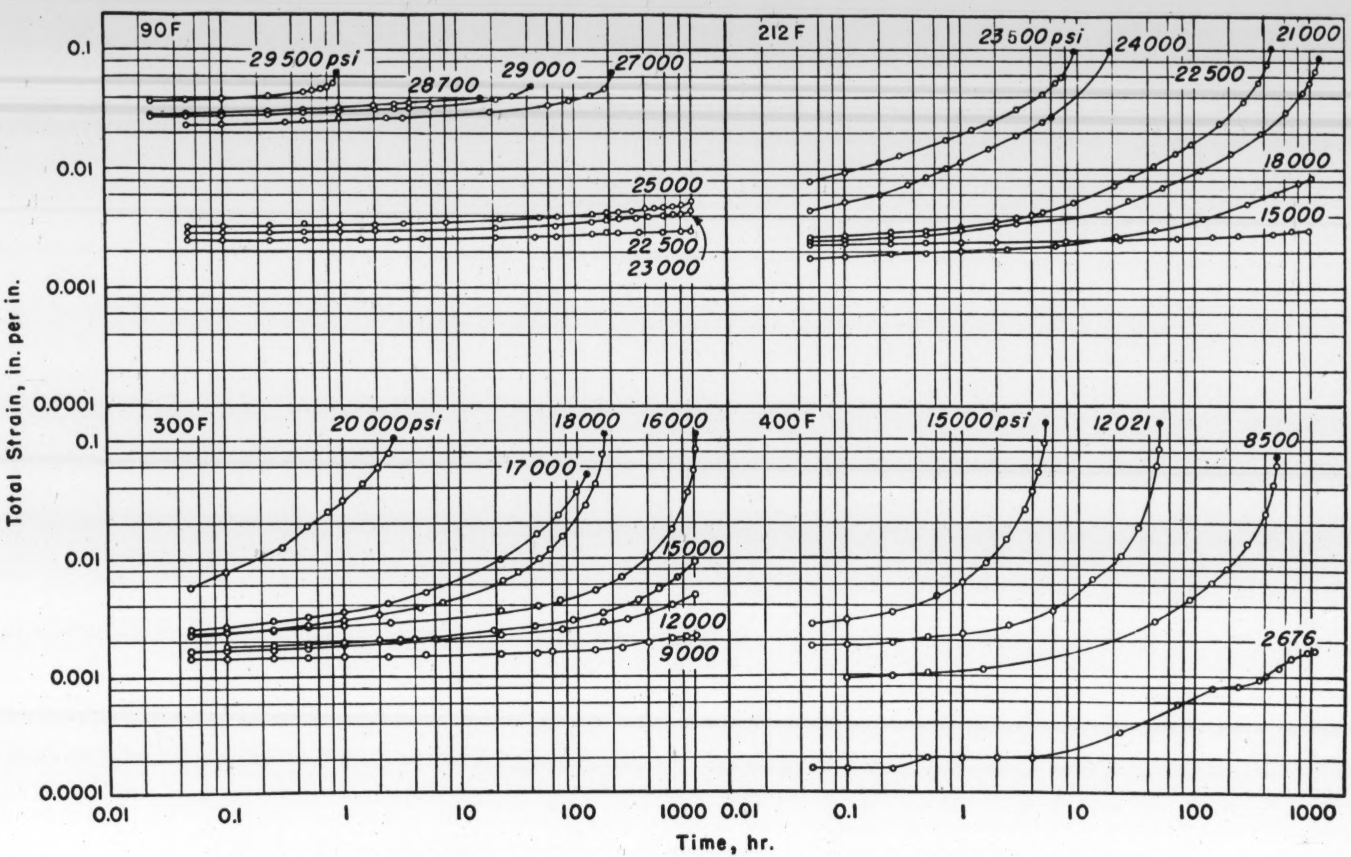


FIG. 3.—Total Creep Strain as a Function of Time for 63S-T5 Material (ASTM Alloy GS10A-T5).

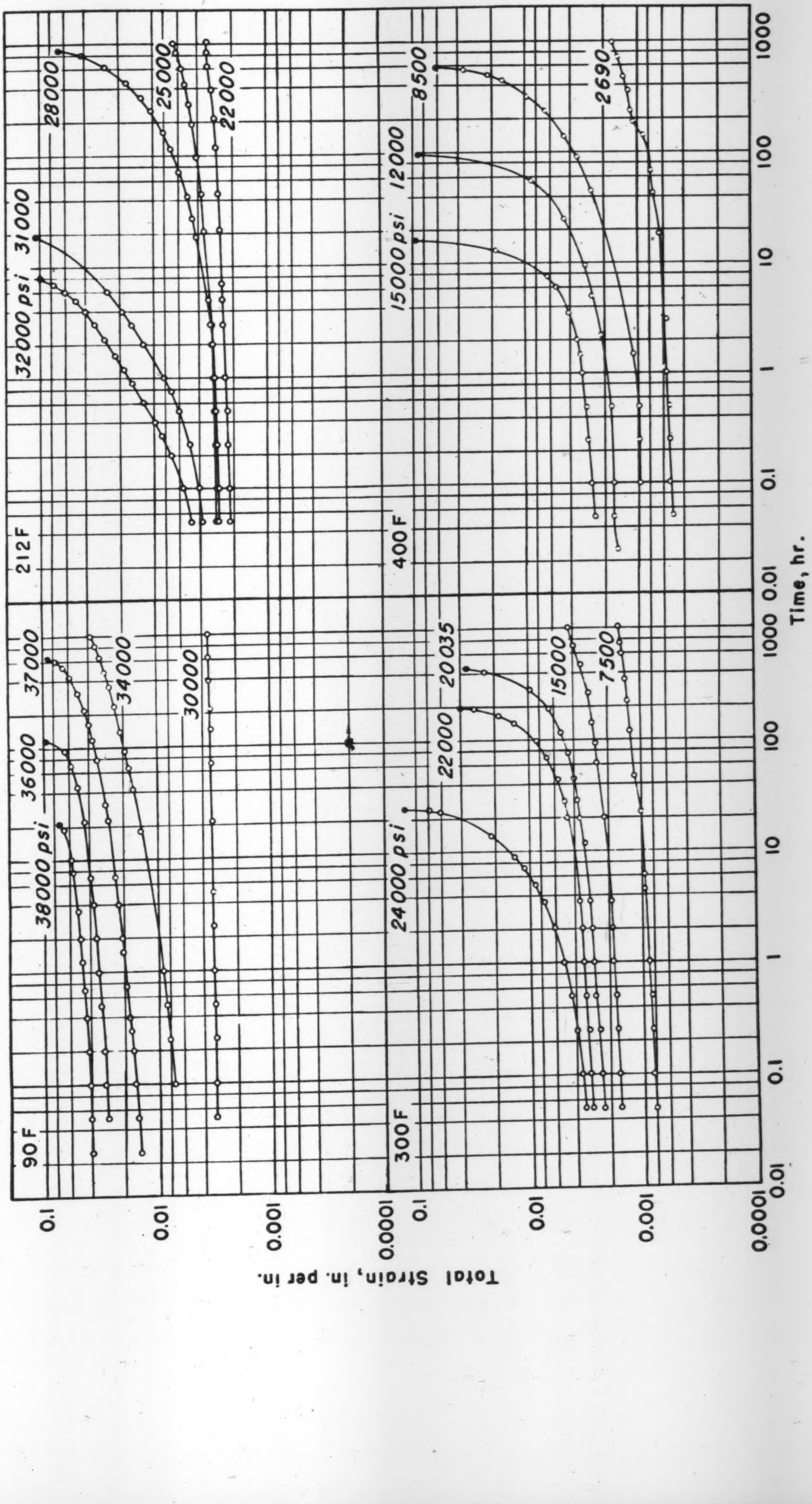


Fig. 4.—Total Creep Strain as a Function of Time for 63S-T6 Material (ASTM Alloy GS10A-T6).

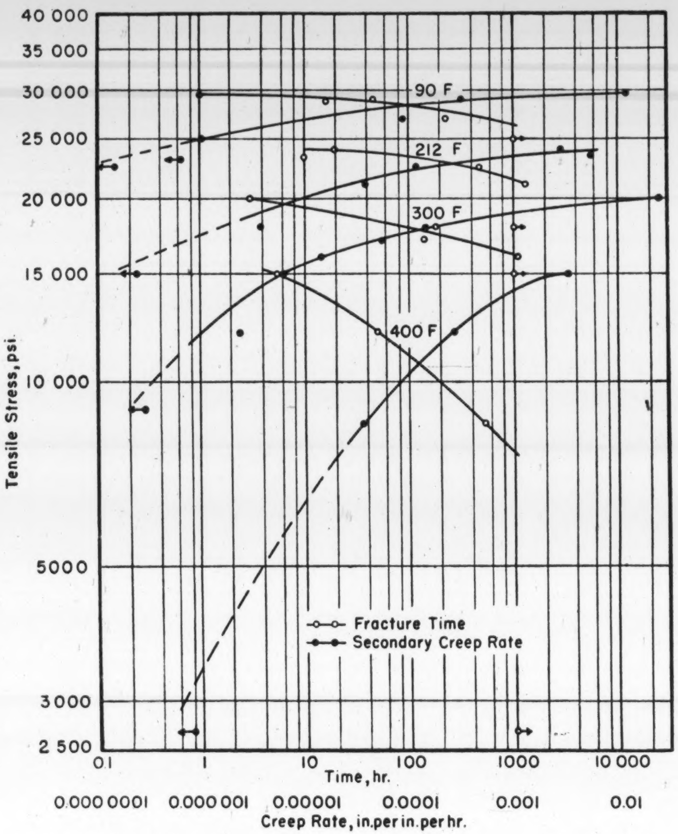


FIG. 5.—Secondary Creep Rate and Stress-Rupture Characteristics for 63S-T5 Material (ASTM Alloy GS10A-T5).

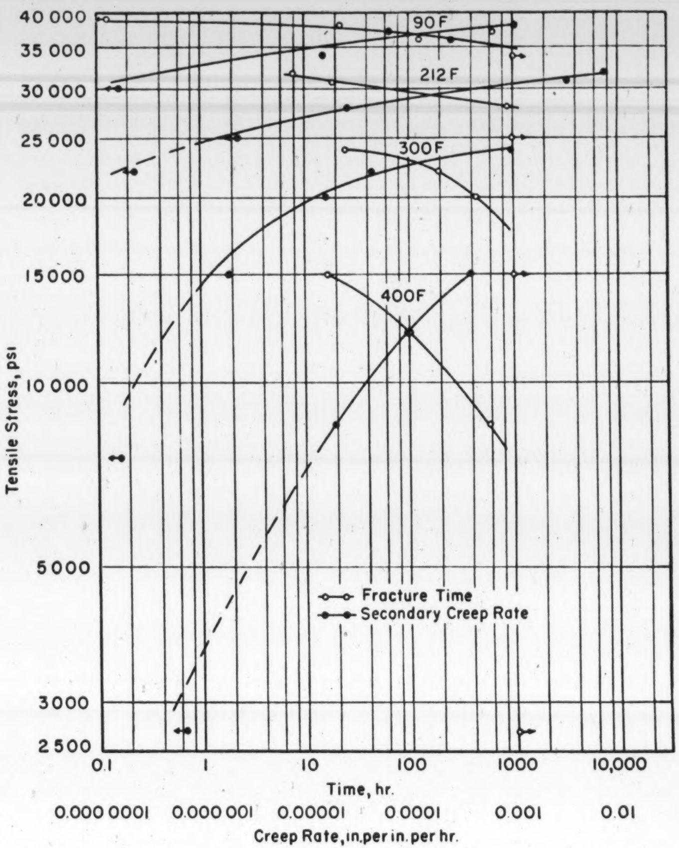


FIG. 6.—Secondary Creep Rate and Stress-Rupture Characteristics for 63S-T6 Material (ASTM Alloy GS10A-T6).

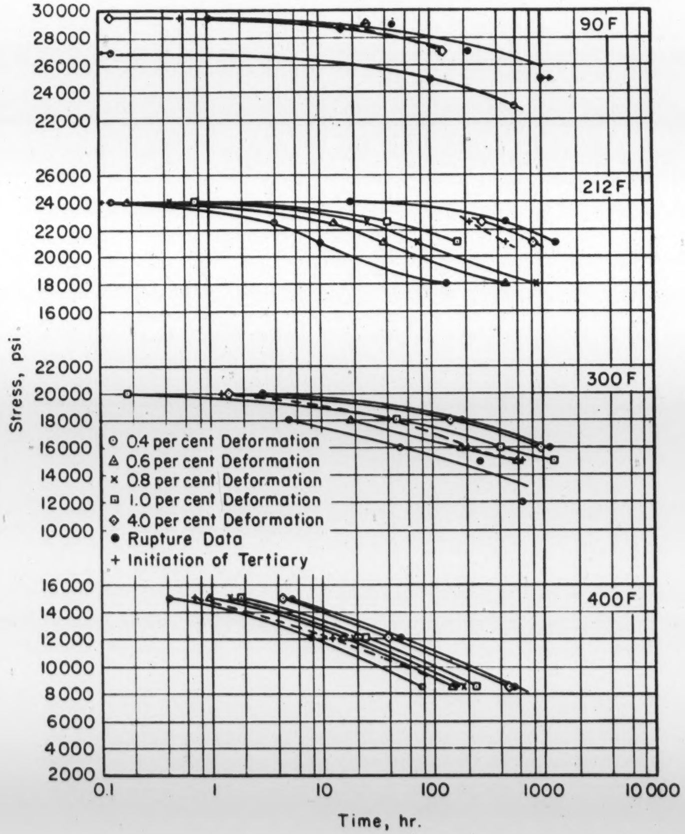


FIG. 7.—Stress *versus* Time for Total Deformation Curves for 63S-T5 Material (ASTM Alloy GS10A-T5).

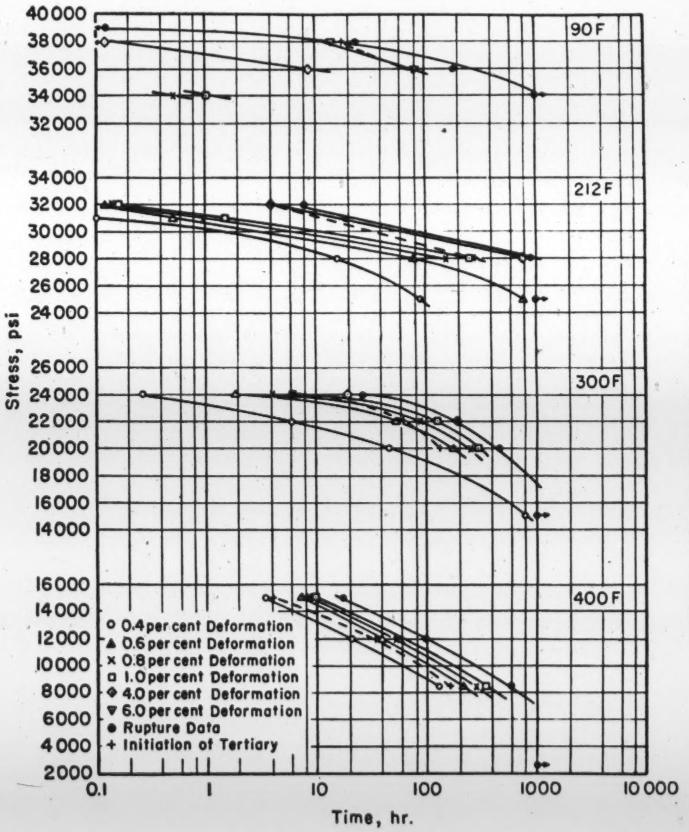


FIG. 8.—Stress *versus* Time for Total Deformation Curves for 63S-T6 Material (ASTM Alloy GS10A-T6).

Tests such as these were discontinued at 1000 hr if rupture had not yet occurred.

Attempts were made to obtain the stress yielding a secondary creep rate of approximately 10^{-7} per hour. In general, however, the very small stresses required to obtain these low strain rates gave a creep curve similar to that illustrated in Fig. 2(*c*). Here the primary stage is so extensive that in most cases the secondary creep rate was not obtainable in a 1000-hr test.

In order to permit rapid comparison of all the original creep data, curves of the logarithm of the creep strain *versus* the logarithm of the time were plotted and are recorded in Figs. 3 and 4.

The effects of stress on the secondary creep rate and on the fracture time are recorded for the two alloys in Figs. 5 and 6 in the conventional log-log coordinates.

For purposes of design it is frequently desirable to know the time under stress for specified total strains. In order to meet this requirement, the data reported in Figs. 7 and 8 were obtained from the original creep results. In addition, the stress *versus* the time to rupture and the stress *versus* the time to initiate tertiary creep are also shown on these graphs.

DISCUSSION OF RESULTS

A comparison of the data recorded in Figs. 5 and 6 reveals that the extruded, air-quenched and artifically aged 63S-T6 material has creep and stress rupture resistance superior to that of the extruded and artifically aged 63S-T5 material. The superior creep resistance of the 63S-T6 material is most pronounced at the lower test temperatures of 90 and 212 F. and the differences between the two tempers of the alloy become smaller at the higher test temperatures of 300 and 400 F. At these higher temperatures the creep resistances of the two tempers of the alloy appear to approach each other at the lower values of the secondary creep rate. The fact that 63S-T6 has a greater creep resistance than 63S-T5, especially at the lower test temperatures of 90 and 212 F., is undoubtedly attributable to the fact that it is in a higher precipitation-hardened state as suggested by the data given in Table II. As higher test temperatures of 300 and 400 F. are used, however, overaging of both materials occurs, especially in the longer tests or lower secondary creep rates. At these temperatures, consequently, the stress increases more rapidly with increasing values of the secondary creep rate than it would in a structurally stable material. As lower secondary creep rates are employed at 400 F, overaging probably causes the structure of 63S-T6 to approach that of 63S-T5. Consequently, at the lower values of applied stress at 400 F., the creep curve of 63S-T6 begins to approach that of 63S-T5.

CONCLUSIONS

1. The 63S-T6 material, which was air quenched from the extrusion temperature and then artificially aged, showed creep properties, superior at all conditions of test, to those of the extruded and artificially aged 63S-T5.
2. At 300 and 400 F., however, the creep properties of 63S-T6 approximated those of 63S-T5 especially at the lower creep rates.

Acknowledgments:

This investigation was sponsored by the Aluminum Company of America. The authors wish to thank the Alcoa Laboratories for preparation of the specimens, for the chemical analyses, and for the determination of the mechanical properties and microstructures.

In addition, they wish to thank Erwin Berliner and M. Kawaguchi for their aid in executing the testing program, and to Mrs. G. Pelatowski they offer their sincere appreciation for the preparation of the numerous graphs which are included in this report.

REFERENCES

(1) A. E. Flanigan, L. F. Tedsen, and J. E. Dorn, "Stress-Rupture and Creep Tests on Aluminum Alloy Sheet at Elevated Temperatures," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 171, p. 213–245. (1947).

(2) O. D. Sherby and J. E. Dorn, "Effect of Annealing on the Creep Properties of 2S-O Alloy," p. 954 of this publication.

(3) J. E. Dorn and T. E. Tietz, "Creep and Stress-Rupture Investigations on Some Aluminum Alloy Sheet Materials," *Proceedings*, Am. Soc. Testing Mats., Vol. 49, p. 815 (1949).

(4) O. D. Sherby and J. E. Dorn, "The Effect of Cold-Rolling on the Creep Properties of Several Aluminum Alloys," *Transactions*, Am. Soc. Metals, Vol. 43, p. 611 (1951).

(5) O. D. Sherby, T. E. Tietz, and J. E. Dorn, "The Creep Properties of Some Forged and Cast Aluminum Alloys," p. 964 of this publication.

(6) O. D. Sherby and J. E. Dorn, "The Creep Properties of Sand Cast Aluminum Materials 319-T71, 319-F and 356-T7," *University of California Institute of Engineering Research Report*, Series 34, Issue 6, October, 1950, Berkeley, Calif.

(7) R. R. Kennedy, "Creep Characteristics of Aluminum Alloys," *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part II, P. 218 (1935).

(8) L. R. Jackson, H. C. Cross, and J. M. Berry, "Tensile, Fatigue and Creep Properties on Aluminum Alloy Sheet at Elevated Temperatures," *Technical Note 1469*, Nat. Adv. Committee for Aeronautics, March, 1948.

[**See Joint Discussion of papers by Sherby, Dorn, and Tietz,** p. 977.—ED.]

EFFECT OF ANNEALING ON THE CREEP PROPERTIES OF 2S-O ALUMINUM ALLOY*

By O. D. Sherby[1] and J. E. Dorn[1]

Synopsis

The effect of stabilizing anneal treatments at 650, 950, and 1150 F on the creep properties of 2S-O aluminum alloy was investigated. Large grains were produced by the 1150 F annealing treatment, but the other two annealing temperatures produced no grain growth and were essentially recovery treatments. The creep rate was found to decrease with increasing temperatures of anneal, irrespective of grain size, within the ranges of stress and temperature which were investigated. The increase in creep resistance by annealing was ascribed primarily to greater grain perfection and not to grain size *per se*. A theory, based on dislocations, is presented in qualitative agreement with the experimental results.

Extensive data are now available on the creep properties of many aluminum alloys (**1** to **8**)[2]. No systematic investigation, however, has yet been reported on the effect of a stabilizing anneal or grain size on the creep behavior of aluminum. Recent investigations (**7**) have demonstrated that the secondary creep rates of some aluminum alloys are sensitive to cold rolling. Whereas cold rolling reduces the secondary creep rates for high stresses at 90 to 212 F, it increases the secondary creep rates for low stresses at 300 to 400 F. A reversal of this trend might be expected when commercial stretcher-straightened or roller-leveled sheet is given a stabilizing anneal.

It is well known that grain size has an important effect on the creep properties of metals. McKeown (**9**) has demonstrated that coarse-grained specimens of lead have superior creep resistance to fine-grained specimens. In fact his data reveal that the secondary creep rate increases almost linearly with the reciprocal of the mean grain diameter. In a more extensive investigation, Clark and White (**10**) have shown that coarse-grained specimens of several copper-zinc-tin alloys have superior creep resistance at elevated temperatures, whereas fine-grained specimens of these alloys are more creep resistant at lower temperatures. Furthermore, the secondary creep rates of the coarse-grained specimens increase more rapidly with increasing stress

TABLE I.—ANNEALING TREATMENTS.

	Heat Treatment: Annealed for 1 hr at Temperature Shown and Cooled in Air	Grains per Centimeter	
		Width	Thickness
A	650 F	200	240
B	950 F	200	240
C	1150 F	3	10

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* Presented at the Fifty-fourth Annual Meeting of the Society, June 18-22, 1951.

[1] Research Engineer and Professor of Physical Metallurgy respectively, University of Calif. Berkeley, Calif.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 963.

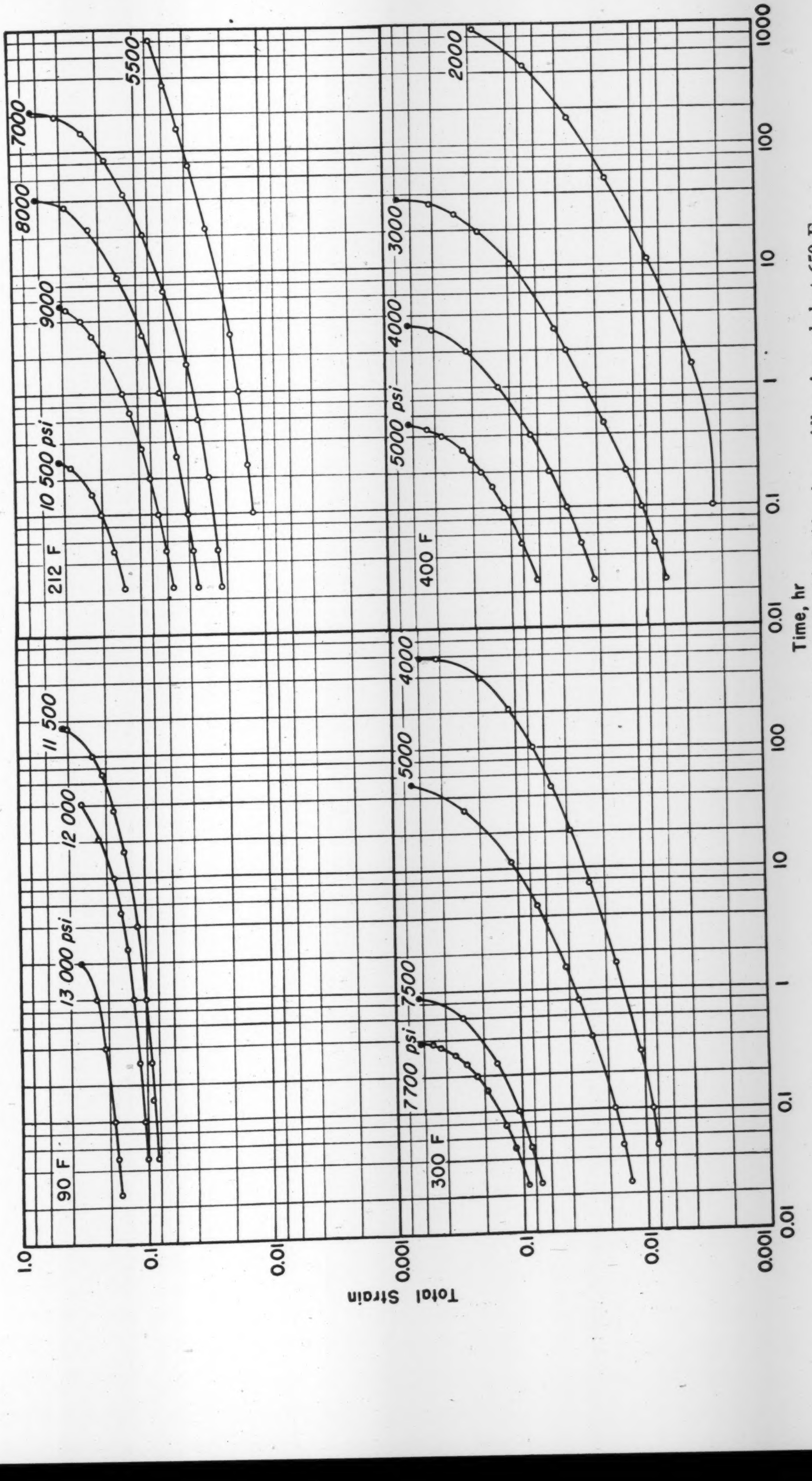

FIG. 1.—Total Creep Strain as a Function of Time for 2S-O Aluminum Alloy Annealed at 650 F.

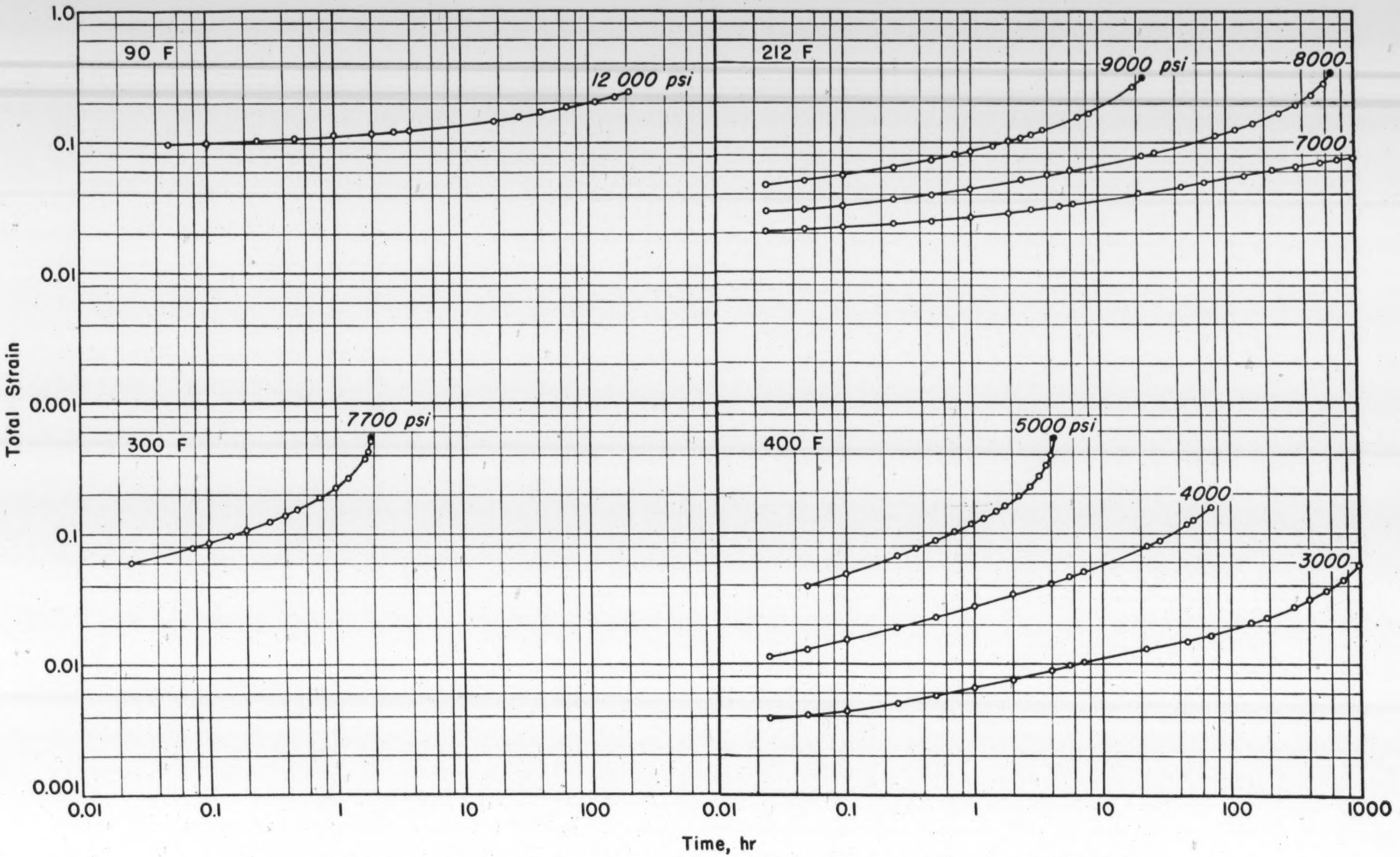

FIG. 2.—Total Creep Strain as a Function of Time for 2S-O Aluminum Alloy Annealed at 950 F.

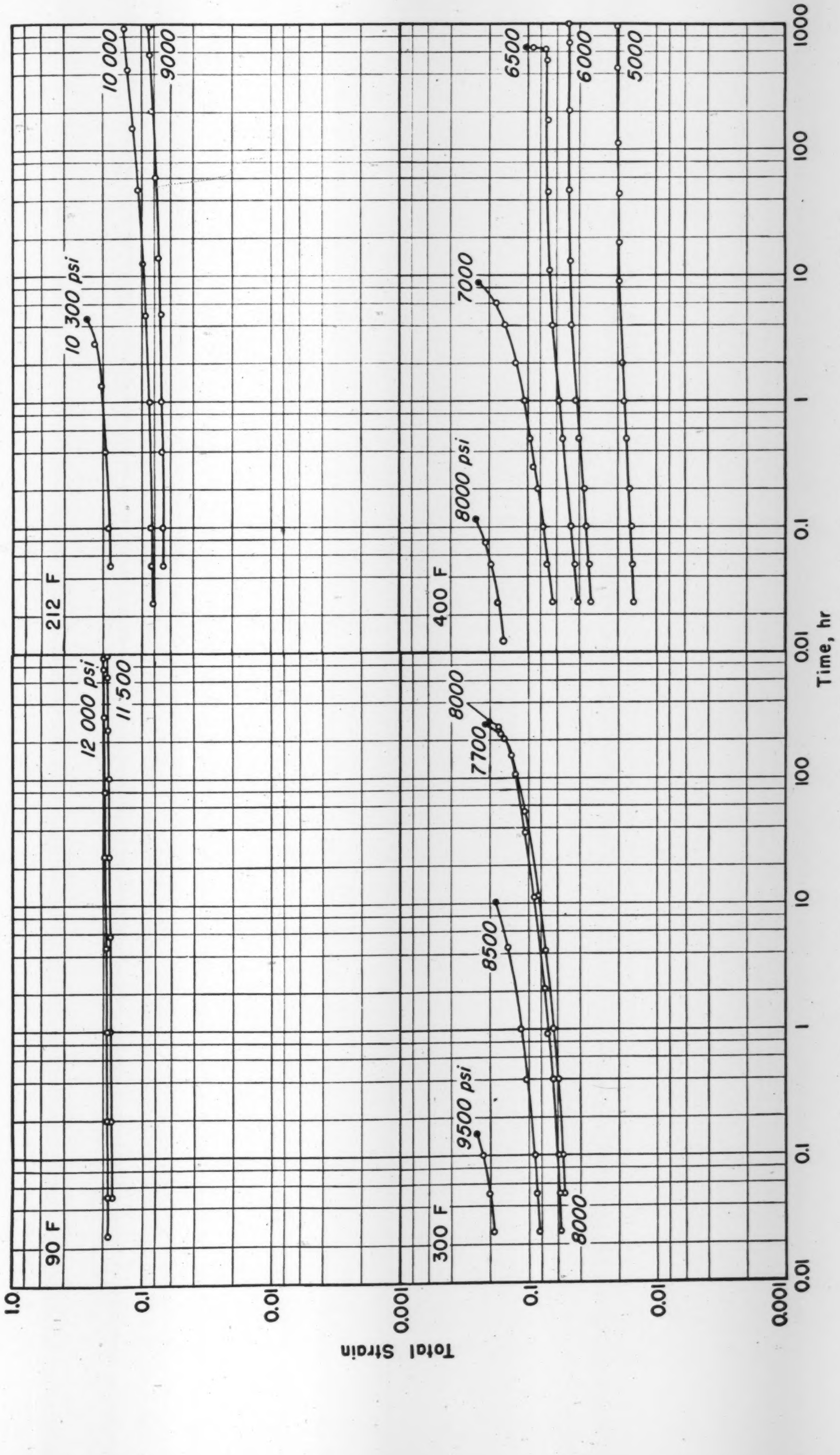

FIG. 3.—Total Creep Strain as a Function of Time for 2S-O Aluminum Alloy Annealed at 1150 F.

than the secondary creep rates of the fine-grained specimens. Consequently, the superior creep resistance of the coarse-grained specimens was observed at the higher creep temperatures and the lower ranges of secondary creep rates. Additional data on the effect of grain size on the creep resistance of metals appear, in general, to confirm the observations of Clark and White. The following report is based on preliminary investigations on the effect of annealing and grain size on the creep properties of commercial 2S-O aluminum alloy.

MATERIAL FOR INVESTIGATION

Commercial stretcher-straightened 2S-O aluminum sheet was used throughout this investigation. In order to study the effect of annealing and grain size, specimens from the same sheet were given one of the three heat treatments recorded in Table I. Treatments A and B were essentially recovery treatments, following which the 2S-O alloy retained its original grain size. Any distinction between the creep behavior of the specimens that were given treatment B and those that were given treatment A is therefore largely attributable to the more complete recovery of the specimens subjected to treatment B. The specimens that were given treatment C, however, experienced appreciable grain coarsening. Consequently, two factors, grain size and a higher degree of lattice perfection, might contribute to any differences between the creep properties of specimens that were subjected to treatment C as contrasted to those that were given treatment B.

All of the creep specimens were selected with their tensile axes in the rolling direction. The specimen design, creep testing equipment, and the methods of analyses employed in this investigation were identical with those which have already been reported in the literature (3).

EXPERIMENTAL RESULTS

In order to facilitate comparisons of the creep behavior of the materials under test over wide ranges of the variables, all of the creep curves were plotted on logarithmic coordinates as shown in Figs. 1 to 3. The solid symbol at the terminus of some of the creep curves represents the point of rupture.

For the purpose of analysis, however, all of the creep curves were also plotted on Cartesian coordinates. A typical creep curve of this type is shown in Fig. 4. Immediately after application of the load an initial strain, ϵ_0, was obtained. This was followed by a transient stage of creep during which the creep rate decreased continuously with time until the minimum creep rate, $\dot{\epsilon}$, characteristic of the secondary stage of creep, was obtained. Subsequently, the creep process entered the tertiary stage where the creep rate continuously accelerated until finally the specimen ruptured.

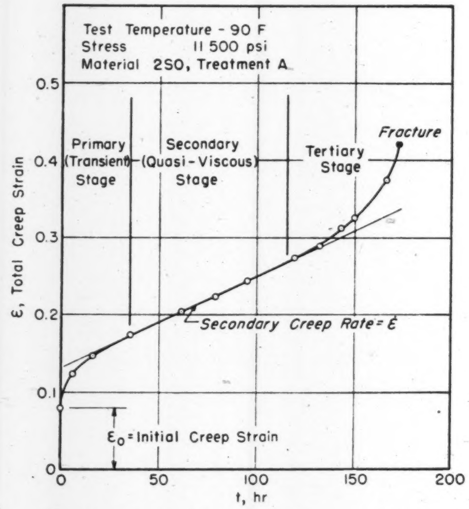

FIG. 4.—Typical Creep Curve for 2S-O Aluminum Alloy Sheet.

The effect of stress on the secondary creep rates and the times to rupture for the three materials under test are recorded in Figs. 5 and 6. At 90 F the secondary creep rates of 2S-O for treatments B and C were so sensitive to the applied stress that only a few data could be obtained for analysis. Rather complete data, however, were obtained for all three test materials at 212 and 400 F. In addition, sufficient information was obtained at 300 F to illustrate that the same general trends of the data were also valid at this intermediate temperature.

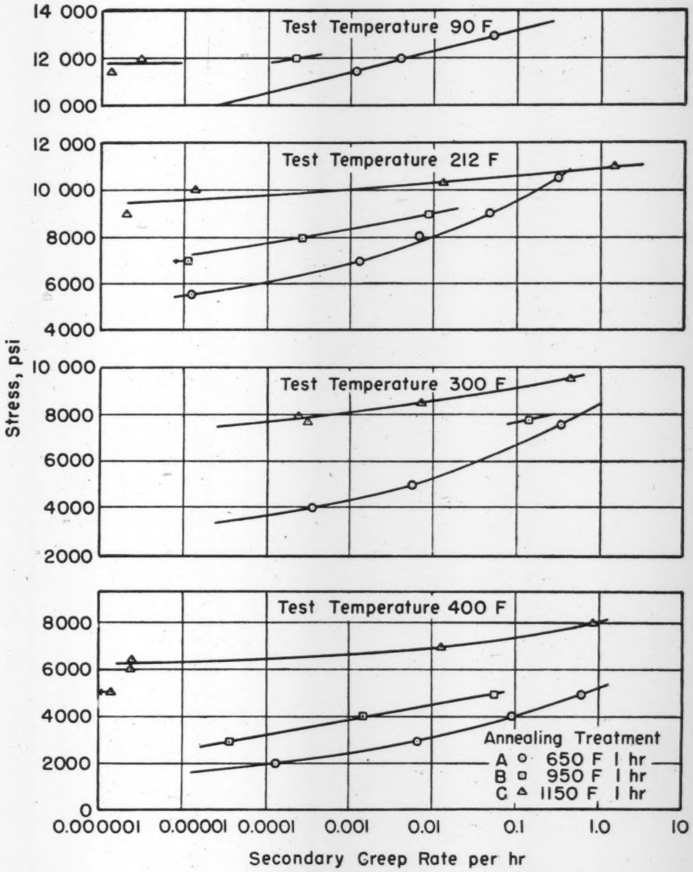

FIG. 5.—Effect of Annealing on the Stress *versus* Secondary Creep Rate Curves for 2S-O Aluminum Alloy.

DISCUSSION OF RESULTS

The data presented in Fig. 5 clearly reveal that the creep resistance of the coarse-grained material that was given treatment C was invariably superior to that for the finer-grained materials which were given treatments A or B. On the other hand, the stress increased most rapidly with increasing secondary creep rates for the material which was given treatment A, whereas the stress increased least rapidly with increasing secondary creep rates for the coarse-grained material that was given treatment C. The secondary creep rates for the material given treatment B were intermediate to those for the materials which were given treatments A and C for all conditions of test that were investigated.

The fact that the slope of the stress *versus* secondary creep rate curve was greatest for the material that was given treatment A suggests that this curve might actually cross those for materials given treatments B and C at some relatively high secondary creep rate. That this inversion actually occurs is indicated by the data of Fig. 5 at 212 F. where the curve for the material that was given treatment A intersects the curve for the material that was given treatment C at a secondary creep rate of about 0.6 per hour and a stress of about 10,800 psi. Furthermore, the data of Fig. 5 also indicate that this inversion in the creep resistance is displaced to higher secondary creep rates as higher test temperatures are employed.

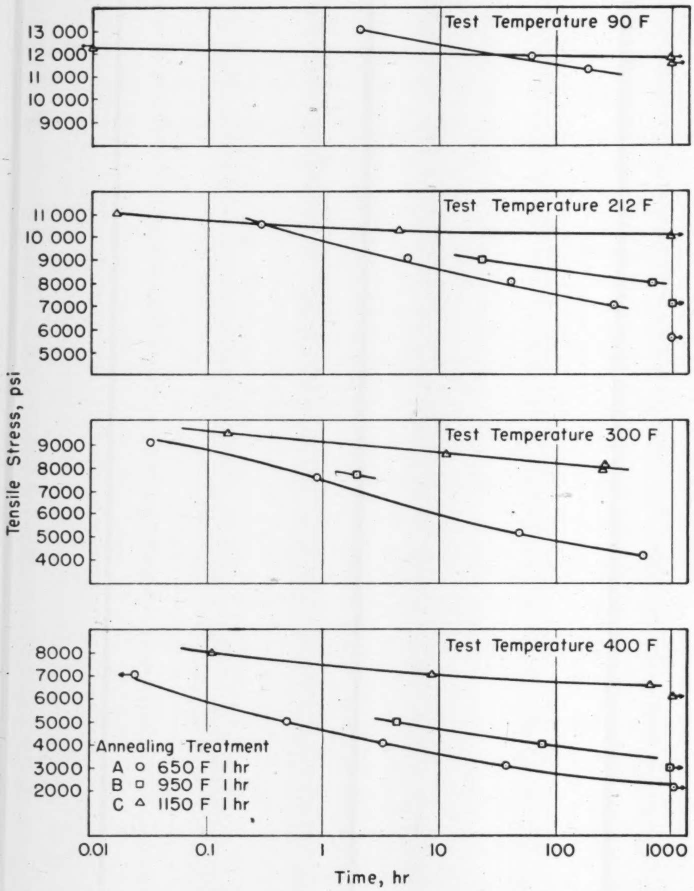

FIG. 6.—Effect of Annealing on the Stress *versus* Time to Fracture Curves for 2S-O Aluminum Alloy.

If the pronounced differences in the creep behavior of the materials that were given treatments B and C were exclusively attributable to the larger grain size of the material subjected to treatment C, the above data would be in complete harmony with the conclusions of Clark and White on the effect of grain size on the creep properties of some copper-zinc-tin alloys. But the same trends are also evident between materials given treatments A and B wherein the grain size was unaltered. Consequently, recovery treatments have the same general effects on the creep properties as have been previously attributed to grain coarsening.

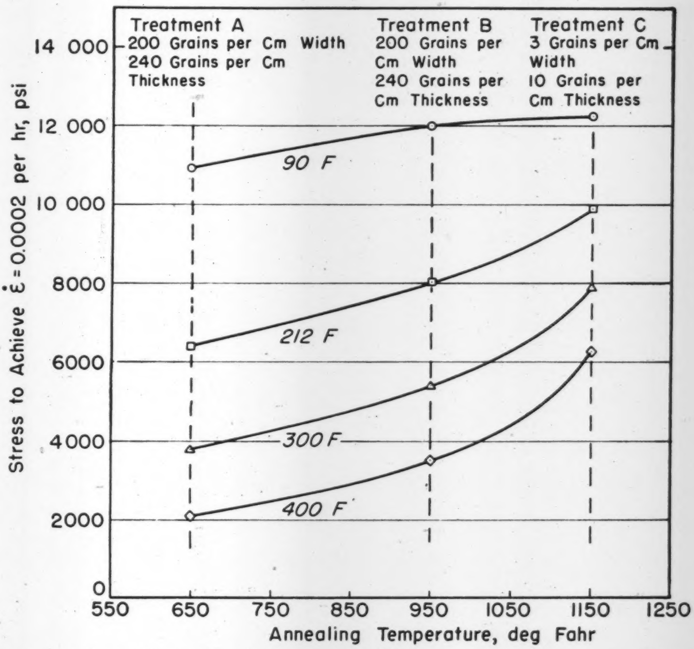

Fig. 7.—Effect of Annealing Temperature on the Stress to Achieve a Secondary Creep Rate of 0.0002 in. per hr. at Various Creep Test Temperatures for 2S-O Alloy.

Additional evidence on the analogous effects of recovery and grain coarsening on the creep properties of materials is shown by the stress-rupture data recorded in Fig. 6. At 400 and 300 F the coarse-grained material which was given treatment C exhibits the longest time to rupture, the material which was given treatment B has an intermediate rupture time, and the material which was given treatment A has the shortest rupture time. At 212 and 90 F these same trends are observed for the tests conducted at the lower stress levels. But, for the higher stress tests, an inversion of this order of resistance to rupture occurs, the material which was given treatment A being most resistant to stress rupture and the material which was given treatment C being least resistant to stress rupture in this range. Consequently, annealing improves the creep resistance of 2S-O alloy at high temperatures and over the lower ranges of secondary creep rates in a manner which is qualitatively reciprocal to the previously observed effect of cold rolling on the creep resistance of aluminum alloys (7).

The creep resistance of stretcher-straightened 2S-O alloy is improved by annealing when the annealing induces only recovery in the absence of grain growth as well as when the annealing causes grain growth. The question then arises as to whether all previously reported observations on the effect of grain size on the creep resistance of metals is attributable to the grain size or whether it is wholly or partially attributable to the greater grain perfection resulting from the more complete recovery of dislocations when the material is heated to the higher temperatures at which grain coarsening occurs. Unfortunately the limited data now at hand do not permit an unambiguous decision on this interesting question. The data shown in Figs. 5 and 6, however, suggest that the creep behavior of the test materials varies in a uniform way with the annealing treatment to which they were subjected. This regular trend is shown more clearly in Fig. 7 where the stress to achieve a secondary creep rate of 0.0002 per hour is plotted as a function of the annealing temperature. Although a definitive answer as to whether grain size also has an effect on the creep resistance would require an additional set of creep data for materials subjected to grain growth by annealing between 950 and 1150 F, the limited data of Fig. 7 appears to vary uniformly with the annealing temperature. This fact suggests that perhaps the higher creep resistance of the coarser-grained material might actually be primarily attributable to greater lattice perfection rather than the grain size *per se*. This thought is in qualitative harmony with the prevalent concept of the rôle of dislocations in the processes of creep.

Due to their quasi-viscous nature, dislocations become highly mobile under stress at elevated temperatures. Cold rolling or stretcher-straightening introduces dislocations in the lattice. At low temperatures these dislocations are not easily activated and deformation proceeds with the generation of many new dislocations. The interaction energy between the dislocations results in strain hardening, and the most resistant material to deformation is that which contains the largest number of dislocations. According to Burgers' model, the grain boundary consists of a series of dislocations. At low temperatures such grain boundary dislocations would react with the dislocations migrating through a grain in such a way as to increase the deformation strength. Consequently, at low temperatures fine-grained metals should be more resistant to deformation than coarse-grained metals of the same lattice perfection. It is quite likely that this factor is of major importance in accounting for the well-known improved tensile properties resulting from grain refinement of many metals.

As the test temperature is increased, however, the dislocations, as a result of their quasi-viscous nature, acquire greater mobility. Consequently, those treatments, such as cold rolling or stretcher-straightening, that introduce dislocations result in materials having lowered creep resistance. Annealing for recovery reduces the number of dislocations either by the process of mutual annihilation or by migration to the grain boundaries. In addition, recovery results in the accumulation of dislocations at sites known as sub-grain boundaries. The reduced number of dislocations results in slower secondary creep rates over those regions of temperature and strain rate where the quasi-viscous nature of the dislocations is the predominant factor that contributes to creep straining. Annealing at higher temperatures, where grain coarsening occurs in addition to recovery, results in additional reductions in the number of dislocations both in the crystallites and in the grain boundary area per unit volume of the metal. Thus, the effect of grain coarsening as well as recovery results in the reduction of the density of dislocations in the metal. Consequently, the effect of grain coarsening on the creep properties of metals should be qualitatively analogous to the effect of recovery alone in spite of minor geometrical differences between the two processes Accordingly it is anticipated that a rather continuous improvement in the high-temperature creep resistance will occur with increasing annealing temperature, and, as shown in Fig. 7, no sharp break in the creep properties will occur as a function of annealing between those temperatures which result solely in recovery and those which also induce grain growth. A minor second order change in the creep properties as a function of annealing temperature at the temperature at which grain growth is initiated, however, is not yet disqualified; this effect is not unanticipated in view of the geometrical distinctions between the processes of recovery of dislocations in the crystallites as contrasted with the removal of dislocations by grain growth.

CONCLUSIONS

1. A stabilizing anneal, which introduces no change in grain size, increases the creep properties of 2S-O aluminum alloy over the ranges of temperatures and strain rates of interest in creep testing. Higher annealing temperatures that cause grain growth induce further resistance to creep and stress rupture.

2. The data obtained suggest that the improved creep resistance generally reported for coarse grained materials at elevated temperatures might be attributable to greater grain perfection rather than the grain size *per se.*

3. The experimental results appear to be in qualitative agreement with the present concept of the rôle of dislocations in the processes of creep.

REFERENCES

(1) R. R. Kennedy, "Creep Characteristics of Aluminum Alloys," *Proceedings*, Am. Soc. Testing Mats., Vol. 35, p. 218 (1935).

(2) "Some Mechanical and Physical Properties of Hindumium," *Research Report No. 56*, Slough, Buckingham, England, High Duty Alloys, Ltd. (1943).

(3) A. E. Flanigan, L. F. Tedsen, and J. E. Dorn, "Stress Rupture and Creep Tests on Aluminum Alloys Sheet at Elevated Temperatures," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 171, p. 213 (1947).

(4) L. R. Jackson, H. C. Cross and J. M. Berry, "Tensile, Fatigue and Creep Properties of Forged Aluminum Alloys at Temperatures up to 800 F," *Technical Note 1469*, Nat. Advisory Committee on Aeronautics, March, 1948.

(5) J. E. Dorn and T. E. Tietz, "Creep and Stress Rupture Investigation on Some Aluminum Alloy Sheet Metals," *Proceedings*, Am. Soc. Testing Mats., Vol. 49, p. 815 (1949).

(6) O. D. Sherby, T. E. Tietz, and J. E. Dorn, "The Creep Properties of Some Forged and Cast Aluminum Alloys," see p. 964 of this publication.

(7) O. D. Sherby and J. E. Dorn, "The Effect of Cold Rolling on the Creep Properties of Several Aluminum Alloys," *Transactions*, Am. Soc. Metals, Vol. 43, p. 611 (1951).

(8) O. D. Sherby and J. E. Dorn, "Creep Properties of Two Tempers of 63S Extruded Aluminum Alloy," see p. 945 of this publication.

(9) J. McKeown, "Creep of Lead and Lead Alloys. I, Creep of Virgin Lead," *Journal*, Inst. Metals, Vol. 60, p. 201 (1937).

(10) C. L. Clark and A. E. White, "Creep Characteristics of Metals at Elevated Temperatures," *Transactions*, Am. Soc. Metals, Vol. 21, p. 12 (1933).

[See Joint Discussion of papers by Sherby Dorn, and Tietz, p. 977.—ED.]

THE CREEP PROPERTIES OF SOME FORGED AND CAST ALUMINUM ALLOYS*

By O. D. Sherby,[1] T. E. Tietz[1] and J. E. Dorn[2]

Synopsis

The creep and stress-rupture properties of the following aluminum alloys were obtained for 90, 212, 300, and 400 F up to 1000 hr rupture time:

Forgings: B18S-T61, 18S-T61, A51S-T6
Permanent-mold castings: 355-T71, A132-T551, 333-T533
Sand Castings: 355-T71, 142-T77, A355-T51

The above sequence of alloys in each group is the sequence of decreasing resistance to creep and stress-rupture over the range of conditions which were investigated. Although the permanent-mold casting 355-T71 had higher creep and stress-rupture resistance than the sand-casting alloy 355-T71 of the same composition at 90 to 300 F, the difference between the two alloys was negligible at 400 F, suggesting that at yet higher temperatures the sand-casting alloy might exhibit superior creep properties to the permanent-mold alloy. It is believed that these observations are attributable to the coarser grain size and dispersion of intermetallic compounds in the sand-cast material.

Over the past few years many engineers have expressed increasing interest in the use of aluminum alloys in elevated temperature service. Creep and stress-rupture data are now available for some sheet materials in the precipitation hardened state,[3] annealed and cold-rolled conditions[4] as well as for a few casting alloys.

The need for additional data on the creep and stress-rupture properties of other standard aluminum alloys prompted the present investigation.

The following materials were tested:

1. Forging alloys:
    - 18S-T61
    - B18S-T61
    - A51S-T6
2. Permanent-mold-casting alloys:
    - 355-T71
    - A132-T551
    - 333-T533
3. Sand-casting alloys:
    - 355-T71
    - 142-T77
    - A355-T51

Creep and stress-rupture properties were evaluated at 90, 212, 300, and 400 F. Stress levels were selected to cause rupture in times ranging from several minutes to about 1000 hr. The following data were obtained for each alloy at the four temperatures:

(*a*) Total creep strain *versus* time,

(*b*) Stress *versus* "time to fracture"

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[3] A. Flanigan, L. Tedsen, and J. E. Dorn, "Stress-Rupture and Creep Tests on Aluminum-Alloy Sheet at Elevated Temperatures," *Technical Publication No. 2033*, Am. Inst. Mining and Metallurgical Engrs. (1946).

[4] J. E. Dorn and T. E. Tietz, "Creep and Stress-Rupture Investigations on Some Aluminum Alloy Sheet Metals," *Proceedings*, Am. Soc. Testing Mats., Vol. 49, p. 815 (1949).

and "secondary creep rate," and

(*c*) Stress *versus* "time for total deformation."

TABLE I.—CHEMICAL COMPOSITION OF ALLOYS INVESTIGATED.

Alloy and Temper	Chemical Composition per cent									
		Silicon	Iron	Copper	Manganese	Magnesium	Chromium	Nickel	Titanium	Zinc
18S-T61	Typical	....	....	4.00	....	0.60	....	2.00	....	....
	Actual[a]	0.56	0.40	3.77	0.02	0.56	0.02	1.96	0.03	....
B18S-T61	Typical	....	....	4.00	....	1.50	....	2.00	....	....
	Actual	0.65	0.36	4.03	0.03	1.50	0.02	2.05	0.03	....
A51S-T6	Typical	1.00	....	....	....	0.60	0.25	....	....	....
	Actual	0.95	0.62	0.22	0.03	0.59	0.26	....	0.06	....
PM355-T71	Typical	5.00	....	1.30	....	0.50	....	....	....	....
	Actual	4.89	0.24	1.29	0.01	0.48	....	0.03	0.13	0.02
PMA132-T551	Typical	12.00	....	0.80	....	1.20	....	2.50	....	....
	Actual	11.85	0.50	1.11	0.04	1.05	....	2.45	0.03	0.03
PM333-T533	Typical	9.00	....	3.50	....	....	....	....	....	....
	Actual	8.46	0.82	3.70	0.35	0.43	....	0.27	0.04	0.45
355-T71	Typical	5.00	....	1.30	....	0.50	....	....	....	....
	Actual	5.07	0.24	1.27	....	0.43	....	0.01	0.13	0.02
142-T77	Typical	....	....	4.00	....	1.50	....	2.00	....	....
	Actual	0.26	0.32	3.92	0.09	1.34	....	1.99	0.20	0.04
A355-T51	Typical	5.00	....	1.40	0.80	0.50	....	0.80	....	....
	Actual	4.83	0.28	1.46	0.88	0.52	....	0.81	0.13	....

[a] Spectrochemical analysis data furnished by the Aluminum Company of America.

TABLE II.—MECHANICAL PROPERTIES OF FORGING ALLOYS.

Alloy and Temper	Properties	Tensile Strength, psi	Tensile Yield Strength, 0.2 per cent offset, psi	Elongation in 2 in., per cent	Compressive Yield Strength, 0.2 per cent offset, psi	Shearing Strength, psi	Brinell Hardness Number
LONGITUDINAL							
18S-T61	Typical[a]	62 000	48 000	12.0	......	......	120
	Actual	60 600	47 000	10.0	47 700	40 500	120
B18S-T61	Typical	60 000	41 000	15.0	......	......	...
	Actual	58 300	41 000	15.0	42 300	38 100	109
A51S-T6	Typical[a]	48 000	43 000	17.0	......	......	100
	Actual	46 000	42 200	19.0	44 100	30 600	96
TRANSVERSE							
18S-T61	Actual	60 600	47 000	9.0	49 300	40 600	...
B18S-T61	Actual	57 500	40 600	11.5	42 000	37 400	...
A51S-T6	Actual	47 500	42 700	14.0	44 800	31 800	...

[a] "Alcoa Aluminum and Its Alloys" (1947).

MATERIALS

All alloys used in this investigation were prepared by the Aluminum Company of America in accordance with accepted standard procedures for melting, casting and forging.

Each of the three sand-casting alloys were cast in six slabs about $\frac{1}{4}$ in. thick, 4 in. wide, and 12 in. long, from a single heat of metal. Plates of PMA132-T551 $\frac{3}{8}$ in. thick, $1\frac{1}{2}$ in. wide and $10\frac{1}{2}$ in. deep and slabs of PM355-T71, PM333-T533 ¼ in. by 1½ in. by 10½ in. were cast in a vertical position from a single heat of metal for each alloy. Radiographic examinations revealed that the castings were uniformly sound, and metallographic examinations revealed that the microstructures were typical for these alloys.

TABLE IV.—GRAIN SIZE.

Type	Alloy	Grains per mm		
		Thickness	Width	Length
Forging	18S-T61	7	3	3
	B18S-T61	18	12	12
	A51S-T6	13	12	3
Sand casting	355-T71	1.0	1.0	1.0
	142-T77	1.8	1.8	1.8
	A355-T51	1.2	1.2	1.2
Permanent mold casting	355-T71	5.0	5.0	5.0

Ingots of the forging alloys were forged in accordance with standard commercial practice into slabs ½ in. thick, 8 in. wide, and 12 in. long. The microstructures in the transverse and longitudinal sections were observed to be typical for these alloys.

TABLE III.—MECHANICAL PROPERTIES OF PERMANENT-MOLD-CASTING AND SAND-CASTING ALLOYS.

Alloy and Temper	Diameter of Tension Specimen, in.	Tensile Strength, psi	Yield Strength, 0.2 per cent offset, psi	Elongation in 4D, per cent	Brinell Hardness Number
PERMANENT-MOLD-CASTING ALLOYS					
355-T71	Typical[a] (0.5)	36 000	31 000	3.0	85
	0.5	38 650	31 500	3.0	86
	0.2	37 800	30 200	2.5	...
A132-T551	Typical[a] (0.5)	36 000	28 000	0.5	105
	0.5	35 300	25 700	1.0	99
	0.2	31 500	24 200	1.2	...
333-T533	Typical[a] (0.5)	32 000	25 000	1.0	100
	0.5	35 100	25 850	1.0	94
	0.2	31 600	25 300	2.5	...
SAND-CASTING ALLOYS					
355-T71	Typical[a] (0.5)	35 000	29 000	1.5	75
	0.5	32 900	27 500	1.3	76
	0.2	32 500	27 300	1.2	...
142-T77	Typical[a] (0.5)	28 000	25 000	2.0	75
	0.5	29 600	22 600	1.4	74
	0.2	29 300	22 700	2.5	...
A355-T51	Typical[a] (0.5)	28 000	24 000	1.5	70
	0.5	27 700	23 700	0.9	71
	0.2	24 000	20 000	2.5	...

[a] "Alcoa Aluminum and Its Alloys" (1947).

The chemical composition of the alloys used in this investigation are compared with their typical compositions in Table I. These compositions were all well within the specification limitations and in most cases agreed well with the typical compositions. In order to characterize further the materials selected for creep testing, the mechanical properties given in Tables II and III were determined. These data show that most of the properties were very nearly those for typical specimens of the alloys to be tested in creep. In the case of the forgings, tension tests were performed on standard ½ in. diameter ASTM specimens.[5] With the cast alloys, the 0.5 in.

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[5] Standard Methods of Tension Testing of Metallic Materials (E 8 - 46), 1949 Book of ASTM Standards, Part 2, p. 993 (1949).

diameter tension specimens were made from separately cast test bars, whereas the 0.2 in. diameter tension bars were machined from the castings. Brinell hardness tests were made using a 500-kg load with a 10-mm ball for 30 sec.

Since grain size is known to have significant effects on the creep properties of alloys, grain size data were also obtained and are recorded in Table IV for all alloys except two of the permanent mold alloys in which cases the grains could not be revealed sufficiently clearly for a grain count.

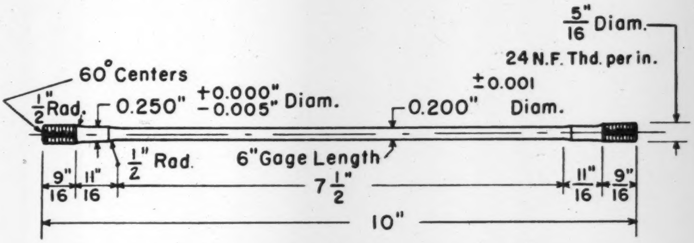

FIG. 1.—Stress-Rupture and Creep Specimen.

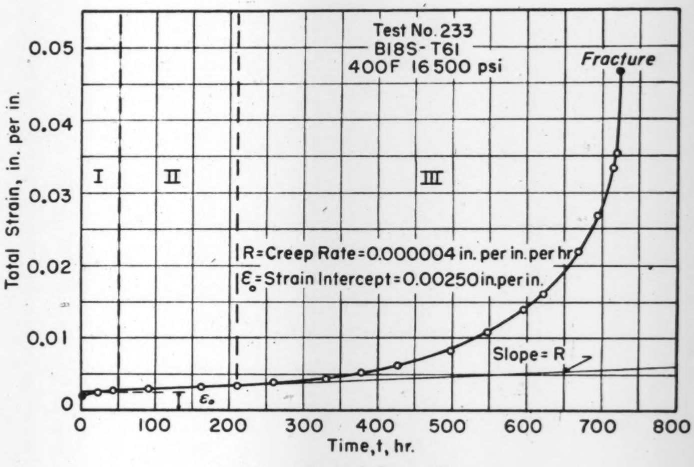

FIG. 2.—Typical Creep Curve.

EXPERIMENTAL TECHNIQUE AND TESTING PROCEDURE

Cylindrical specimens of the design shown in Fig. 1 were used in this investigation. Except for minor changes in the method of gripping the specimens and attaching the extensometers, the testing technique was substantially identical with that which has been described previously.[3, 4]

Loading of the specimen was obtained through 10:1 levers, to which metal containers holding lead shot were attached. The extensometer dials were graduated in divisions equivalent to 0.005 in. change in gage length. Readings were made to one tenth of a division corresponding to a strain of 0.00008. Temperatures for the 400 and 300 F furnaces were maintained constant by boiling glycol-water solutions. Boiling water was used to obtain the 212 F temperature, and a thermostatic control with water to maintain the 90 F temperature.

The specimens were inserted into the

furnace 1 hr prior to application of load in order to permit the specimens to reach the testing temperature. The load was then gradually applied and the extensometer was read on attainment of full load.

EXPERIMENTAL RESULTS AND EVALUATION OF DATA

During the course of a test, strain readings were made at frequent intervals to define complete strain *versus* time curves (creep curves) which were plotted in cartesian coordinates as illustrated in Fig. 2. All of the significant data for creep were obtained from such curves.

The secondary creep rate was obtained by a graphical determination of the slope, R, of the straight-line portion of the creep curve (Range II, Fig. 2).

DISCUSSION OF RESULTS

Stress Versus "Fracture-Time" and "Secondary-Creep-Rate" Curves:

The effect of stress on the secondary creep rate and the time to rupture for the forging alloys are recorded in Fig. 3. Not only are the tensile strengths of 18S-T61 and B18S-T61 superior to those of A51S-T6 but their creep resistance and resistance to stress rupture are also substantially greater. These differences appear to be attributable primarily to differences in composition. Although B18S-T61 has slightly lower yield and tensile strengths than 18S-T61, it nevertheless exhibits superior resistance to creep and stress rupture at all temperatures except perhaps at 400 F. It might be expected that the higher magnesium content of B18S-T61 would cause its resistance to creep to be superior to that of 18S-T61. The trends of the creep properties with increasing temperature strongly suggest, however, that 18S-T61 might be superior to B18S-T61 at some temperature slightly above 400 F. If this superiority of 18S-T61 exists at the higher temperatures, it may be because of its coarser grain size.

The creep properties of the three permanent-mold-casting alloys are recorded in Fig. 4. The first alloy reported, PM355-T71, has the highest resistance to creep and stress rupture; PMA132-T551 has intermediate creep properties, whereas, PM333-T533 has the lowest creep properties. Reference to the mechanical properties given in Table III reveals that this order does not correlate well with either the tensile strengths or the yield strengths of these alloys.

It will be noted that the scatter in the creep data for the permanent-mold alloys is somewhat greater than the scatter obtained for the forging alloys. This scatter is particularly great at 90 F for PMA132-T551 and also PM333-T533. Undoubtedly some of the scatter in these data are attributable to the small diameters (0.2 in.) of the specimens used in this investigation. Any given microstructural variation in this case would occupy a greater percentage of the cross-sectional area of the specimen than in a larger specimen and, therefore, it would exhibit a proportionally greater effect on the creep properties of the smaller specimen. If this is the cause of the scatter in the data, the effect of microstructural variations might be expected to be greater on the stress to rupture than on the secondary creep rate, a deduction which is in agreement with the experimental facts. Furthermore, the scatter should in general be greatest for sand castings, intermediate for the permanent-mold castings and least for the forgings since this is the usual order of increasing microstructural homogeneity. In general this was the order of decreasing scatter which was observed in the present investigation. The reason why the scatter is greater at 90 F than at the higher temperatures is probably associated with the fact that the reduction of stress con-

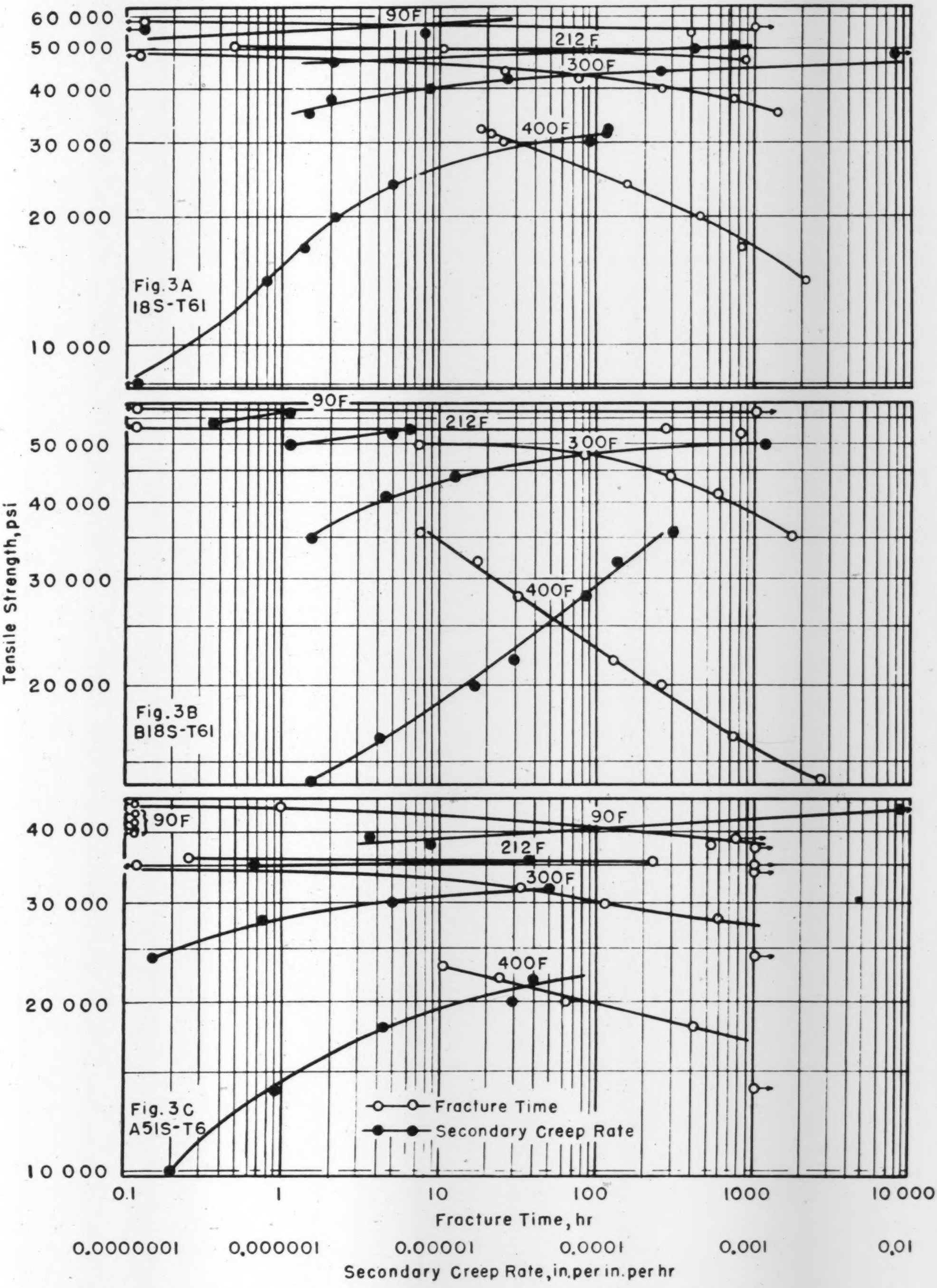

FIG. 3.—Secondary Creep Rate and Stress-Rupture Characteristics for Three Forging Alloys.

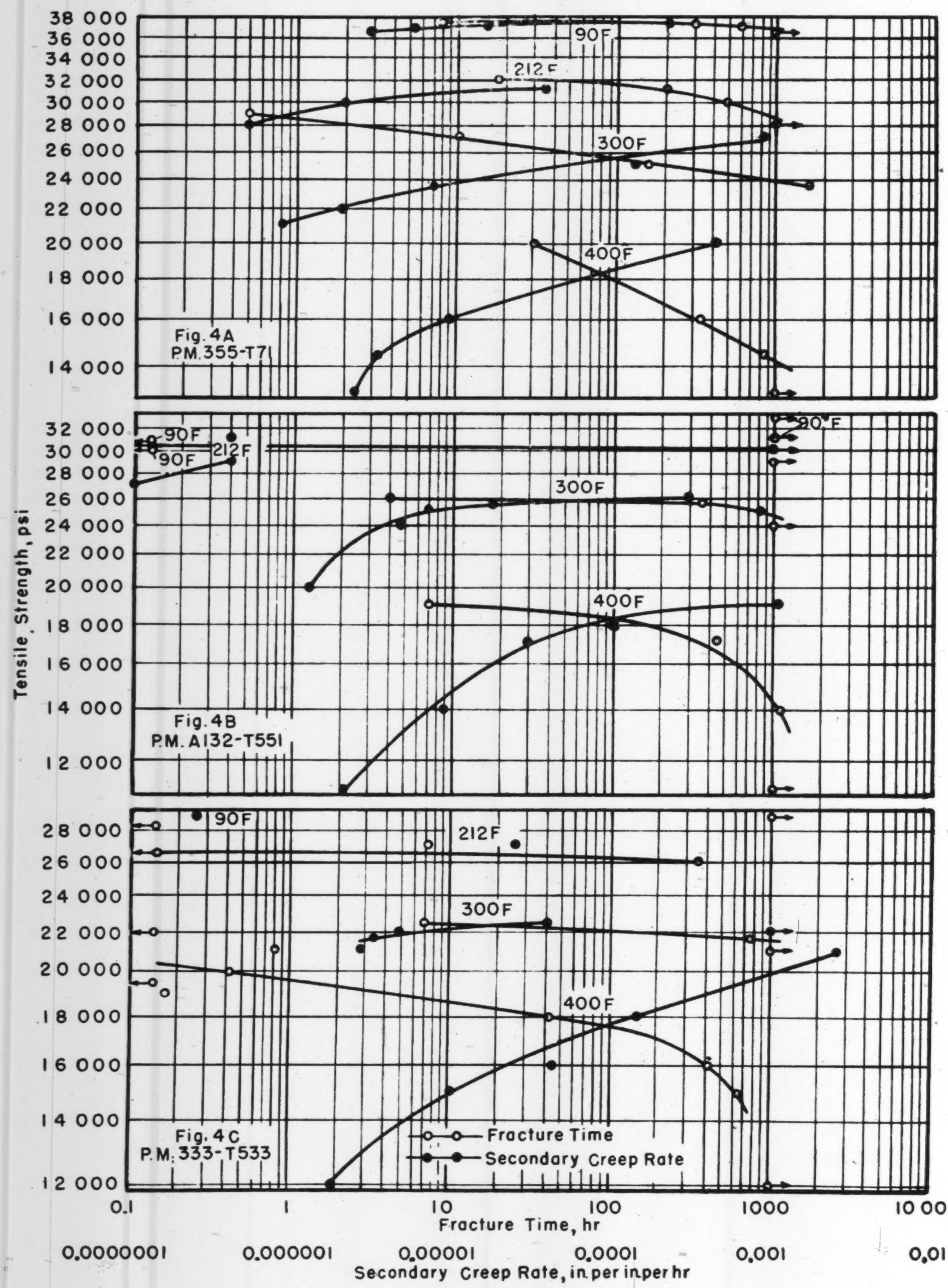

FIG. 4.—Secondary Creep Rate and Stress-Rupture Characteristics for Three Permanent Mold Casting Alloys.

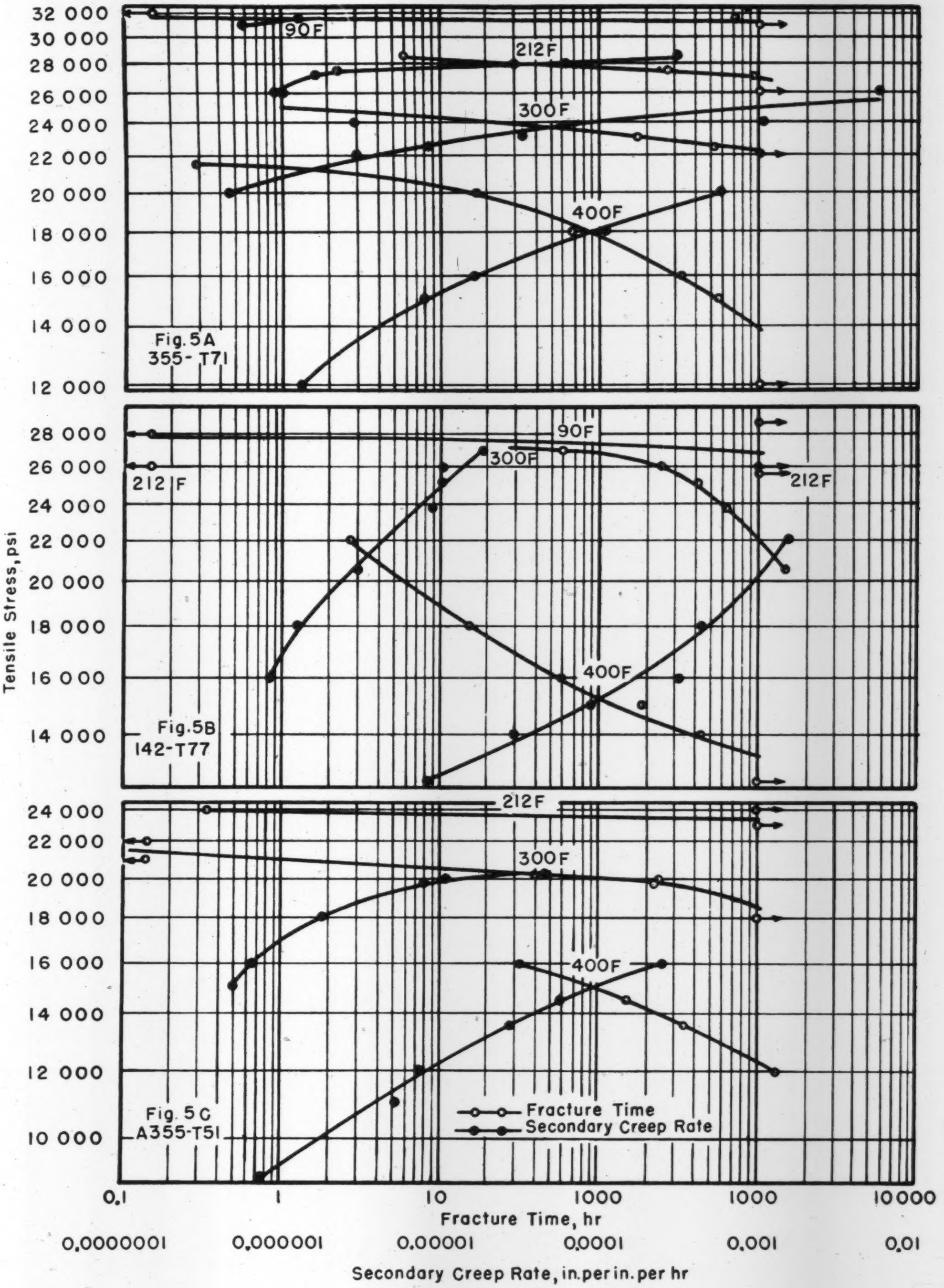

FIG. 5.—Secondary Creep Rate and Stress-Rupture Characteristics for Three Sand-Cast Alloys.
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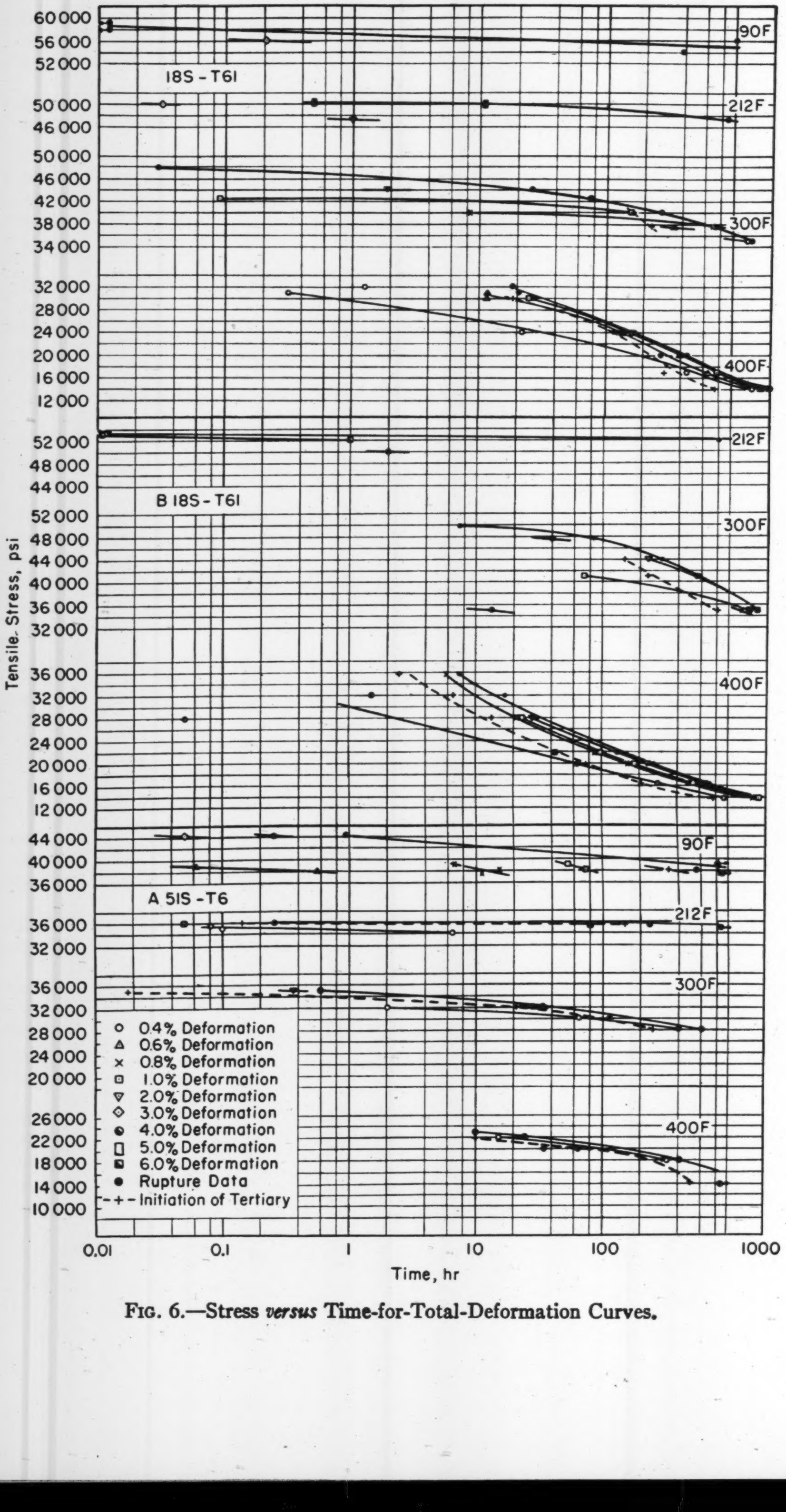

FIG. 6.—Stress *versus* Time-for-Total-Deformation Curves.

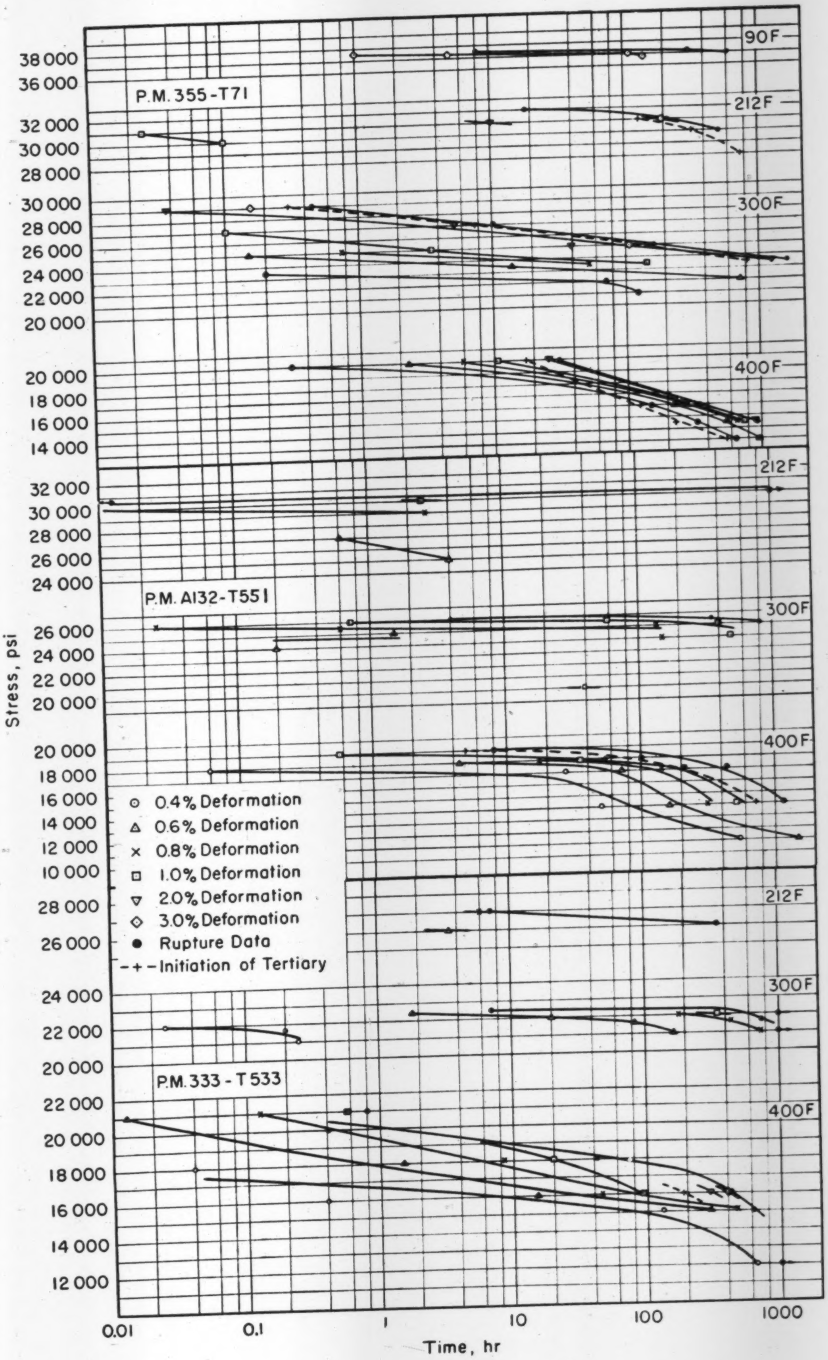

FIG. 7.—Stress *versus* Time-for-Total-Deformation Curves.

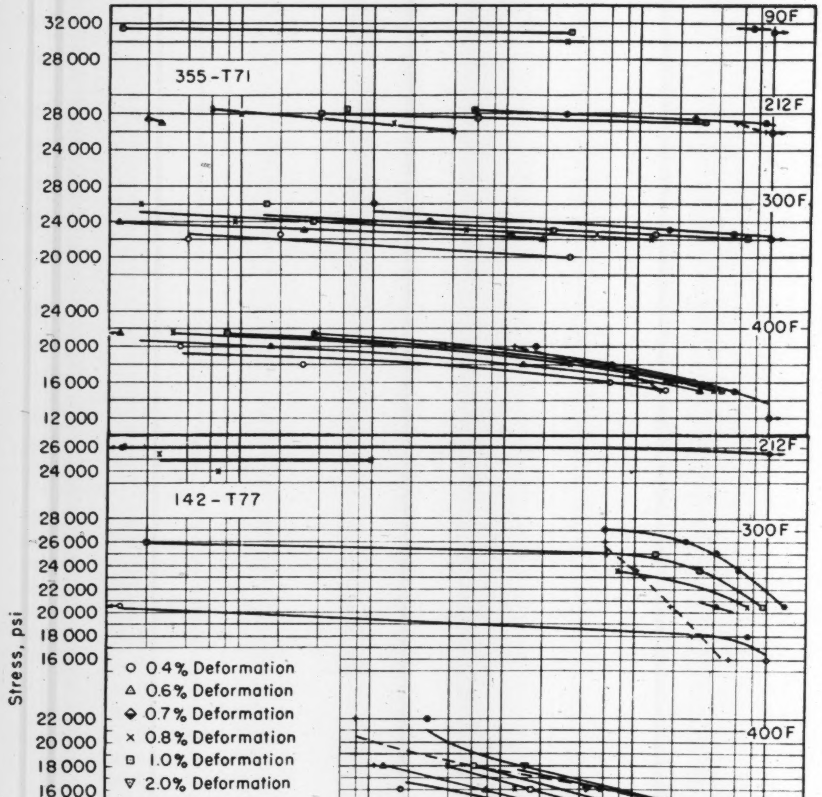

FIG. 8.—Stress *versus* Time-for-Total-Deformation Curves.

centrations about microstructural inhomogeneities is less pronounced at the lower temperatures because of the slower rates of plastic deformation.

The creep properties of the sand-casting alloys are recorded in Fig. 5. The order 355-T71, 142-T77, A355-T51 is that of decreasing creep and stress-rupture resistance. This is also the order of decreasing tensile strength and yield strength.

The permanent-mold alloy PM355-T71 has about the same composition and heat treatment as the sand casting 355-T71. Consequently the differences in the properties must be attributable to the differences in the microstructure induced by the two alternate casting procedures. The higher tensile and yield strength of the permanent-mold casting are probably attributable to the finer dispersion of the microconstituents as well as finer grain size. The same factors are undoubtedly responsible for the superior creep and stress-rupture characteristics of the permanent-mold alloy at temperatures of 90 F to somewhat above 300 F. At 400 F, however, the creep and stress-rupture characteristics of the permanent-mold and sand-cast states of 355-T71 give almost the same creep properties. This trend strongly suggests that the creep properties of the sand casting might become superior to those of the permanent-mold casting at temperatures above about 450 F. It is not yet clear whether this difference is attributable to the coarser dispersion of the intermetallic compounds or the coarser grain size of the sand casting. Recent investigations[6] have established the fact that coarser grained 2S-O alloy has higher creep resistance than finer grained 2S-O alloy at the higher temperatures. It is also possible that the effect of coarser dispersions of intermetallic compounds in aluminum alloys cause effects analogous to those of coarser grain size.

Stress Versus "Time-for-Total-Deformation" Curves:

For purpose of design it is frequently desirable to know the time under stress for specified total strains. In order to meet this requirement the data reported in Figs. 6, 7 and 8 were obtained from the original creep results. In addition the stress-rupture and stress-time to initiate tertiary creep were also reported on these graphs. The latter was included because it might prove more important in design for higher temperature service than merely the time for a specified strain. For example, consider the data recorded in Fig. 6 for 18S-T61 alloy. The design requirements may admit a strain of 0.4 per cent in 1000 hr allowing a stress of about 15,000 psi at 400 F. But under these conditions the material is already strained well within the dangerous tertiary stage of creep. It would appear to be desirable to use a stress of about 12,000 psi which is determined by the initiation of the tertiary stage of creep in order to avoid the dangers associated with the rapid tertiary creep rates.

It was not possible to obtain highly accurate data on the initiation of the tertiary stage of creep and consequently the data refer to the best estimates that could be made on the time for beginning the third stage of creep. Generally the third stage of creep occurs at lower total strains as the temperature increases and as the stress is lowered. For low stresses (that is, long times for creep) the initiation of the third stage of creep appears to be a more appropriate design criterion than the time for a specified strain.

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[6] O. D. Sherby and J. E. Dorn, "Effect of Annealing on the Creep Properties of 2 S-O Alloy," see p. 954 of this publication.

CONCLUSIONS

1. The creep and stress-rupture properties have been determined for the following aluminum alloys over the range of 90 to 400 F:

Forgings: B18S-T61, 18S-T61, A51S-T6.

Permanent-mold castings: 355-T71, A132-T551, 333-T533.

Sand Castings: 355-T71, 142-T77, A355-T51.

The alloys above are arranged in each group in the order of decreasing resistance to stress-rupture and creep.

2. The permanent-mold casting 355-T71 has superior creep resistance to the sand casting of the same composition over 90 F up to about 375 F. It appears that at higher temperatures the sand casting might acquire higher resistance to creep and stress-rupture than the permanent-mold casting.

Acknowledgments:

This investigation was sponsored by the Aluminum Company of America. The authors wish to acknowledge the many important contributions made by the staff of Alcoa Research Laboratories. In particular they thank the Alcoa Laboratories for preparation of the alloys, for the chemical analyses, and determination of the mechanical properties and microstructures as well as for their ever available help and advice throughout the planning and creep testing programs of this investigation.

In addition, they wish to thank Mr. E. Berliner for his aid in executing the testing program, and to Mrs. G. Pelatowski they offer their sincere appreciation for the preparation of the numerous graphs of this report.

JOINT DISCUSSION OF PAPERS BY O. D. SHERBY, J. E. DORN, AND T. E. TIETZ

MR. G. R. GOHN[1] (*presented in written form*).—Creep data of the type presented by the authors of these papers are very useful to the designer. The data would be even more valuable had the tests been continued for a somewhat longer period than the 1000 hr used by the authors in their studies. The longer testing periods are required if significant secondary creep rates are to be obtained. This is evident from the authors' data which indicate that the curves portray the second stage of creep only at the higher stresses. For example, in the first of these three papers the curve portrayed in Fig. 2(*c*) indicates no secondary creep rate.

From the standpoint of the reader, it would be very helpful if the creep characteristics of the various alloys were compared at each of the several test temperatures. If this is done, it will be seen, for example, that the differences in creep rates for the three sand-casting alloys are negligible when tested at 400 F. In the case of the three forging alloys and the three permanent mold casting alloys, there are still significant differences in creep rates at 400 F, but the differences are less in these tests than in the tests made at 300 F.

In the first of the three papers the authors have observed that at 300 and 400 F, the creep properties of alloy 63S-T6 approximate those of the 63S-T5 alloy, especially at the lower creep rates. These observations are in agreement with the findings of J. McKeown and R. D. S. Lushey in England, who in their article on Creep Properties of Some Aluminium Alloys at Temperatures Up to 300 C (572 F)[2] show the results of a series of creep studies on cast and forged aluminum alloys. One extrusion (Alloy 25S) is also included in their work. While the composition of the alloys studied by these two English investigators differ somewhat from the standard alloys used in this country, their findings are believed applicable. On the basis of their tests, McKeown and Lushey reported that, while there are appreciable differences between the creep resistance of various aluminum alloys at lower temperatures, these differences tend to disappear at 300 C. The work reported in the papers just presented here, while not carried to as high a temperature, indicate a similar trend for the creep rates of the ten alloys studied. This trend might have been even more clearly demonstrated had the testing period been long enough to permit the determination of secondary creep rates at all of the stress levels studied.

MR. F. S. GARDNER[3] (*presented in written form*).—This information supplied by the University of California and Alcoa on some aluminum forgings, permanent mold castings, and sand castings supplements their earlier contribution on sheet materials and fortifies the design engineer with hitherto unavailable knowledge. The data at 90 and 212 F are particularly welcome, in view of the increasing pressure to substitute aluminum and its alloys for steel and copper-base alloys. For example, in designs where stress relaxation of an aluminum member must be minimized over a design life of 20 years, the creep and stress for fracture properties must be known if section size (and also cost) is to be kept comparable with the former materials.

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[1] Member of Technical Staff, Bell Telephone Laboratories, Inc., New York, N. Y.

[2] *Metallurgia*, Jan., 1951, p. 15.

[3] Metals Section, Transformer and Allied Product Divs., Apparatus Dept., General Electric Co., Pittsfield, Mass.

Inspection of the secondary creep rate and stress rupture characteristics of aluminum alloys, Figs. 3, 4, and 5 of the third paper shows the two associated lines to approach closely a straight line at the lowest temperature, namely, 90 F. I might reason that these particular lines could be extrapolated to lower stresses with fair assurance. This is on the basis that the observed failure of the straight-line extrapolation at 300 and 400 F is accounable by an "expected" instability at these temperatures, whereas these aluminum materials are not "expected" to be instable at room temperature. The authors comments on this assumption would be appreciated. The values of the elongation to fracture, where available, might cast some light on this.

Table II reports the actual yield strength (0.2 per cent offset) of alloy A51S-T6 to be 42,200 psi. Figure 3 (*c*) indicates that at this stress the time for fracture is only of the order of 20 to 40 hr. This is the only such instance among the materials reported, and I should like to known whether, in general, this is the exception rather than the rule.

MESSRS. O. D. SHERBY, T. E. TIETZ AND J. E. DORN (*authors' closure*).—Mr. Gardner's suggestion that a linear relationship might exist between the logarithm of the applied stress and the logarithm of the creep rate for aluminum alloys at low temperatures should be accepted with caution. Published results on other aluminum alloys[4, 5] appear to indicate that this relationship is curvilinear, and hence too much weight should not be placed on any such correlations. One of the reasons contributing to such apparently linear relationships at ambient temperatures is ascribable to the fact that the creep rate is highly sensitive to small changes in stress; consequently, the slope of the stress-secondary creep rate data is very nearly flat, and the presence of any true curvature in such plots is usually difficult to observe.

Little or no correlation has been found by the authors between the yield strengths of aluminum alloys and their corresponding creep resistance. When the yield strength of a material is near its ultimate tensile strength then such results as in the case of A51S-T6 forging alloy may occur; that is, the material may creep rapidly and fail early if stressed under constant load at its yield strength (0.2 per cent offset). Other aluminum alloys that have been found to behave in a similar manner are 2S, 3S, and 99.6 per cent aluminum in various cold-rolled conditions investigated earlier.[4, 5]

MR. F. M. HOWELL[6].—We have made some creep tests on some of these alloys at Aluminum Research Laboratories, and our results on 18S-T61, B18S-T61 and A51S-T6, the three forging alloys, agree quite satisfactorily with the results reported by the authors. These tests were made at temperatures of 300 and 400 F.

I do want to point out, however, that the area in which we found the most difference is at the lower stresses, where we found lower creep rates than the authors did.

I believe this is in agreement with what Mr. Gohn was speaking of, and also, as Mr. Gardner has found in his tests, our creep rate and time to rupture results approach more closely straight lines than the results reported by the authors. I think that most of the curva-

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[4] J. E. Dorn and T. E. Tietz, "Creep and Stress-Rupture Investigations on Some Aluminum Alloy Sheet Metals," *Proceedings*, Am. Soc. Testing Mats., Vol. 49, p. 815 (1949).

[5] O. D. Sherby and J. E. Dorn, "The Effect of Cold Rolling on the Creep Properties of Several Aluminum Alloys," *Transactions*, Am. Soc. Metals, Vol. 43, p. 611 (1951).

[6] Chief, Mechanical Testing Div., Research Laboratories, Aluminum Company of America, New Kensington, Pa.

ture they have is attributable to the fact that they have relatively high creep rates at the lower stresses.

Our creep rates for such stresses are lower, which tends, of course, to straighten out the lines.

MR. M. J. MANJOINE.[7]—My remarks will be directed toward a standardization in reporting the results of creep-rupture tests.

Many investigators have been reporting their creep-rupture results by using a family of curves which we refer to as "Design Curves." These are curves in which the stress is plotted as a function of time for various criteria of failure. The criteria of failure are usually (*a*) "time to a given total or plastic strain," (*b*) "time to reach the transition from the second to the third stage of creep, and (*c*) "time to rupture."

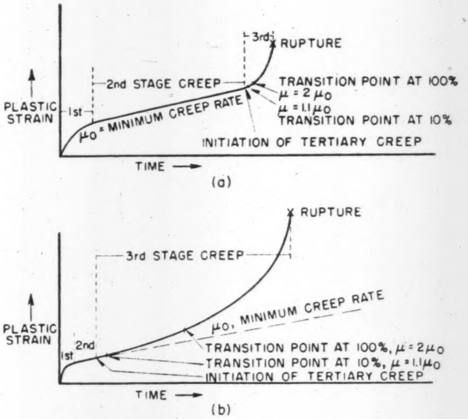

FIG. 1.—Creep Rupture Curves.

There is little controversy as to criteria "*a*" and "*c*," but as yet there is no agreement about criterion "*b*."

The authors identify this point "*b*" as the "initiation of tertiary creep." We call this point the "transition point" and define it as that point in the third stage of creep at which the creep rate has increased 10 per cent over the minimum rate.[8] If the "transition point" is considered as a limit of the useful life of a material, then the above definition is not adequate. For example, in Fig. 1, two general types of creep-rupture curves are drawn. Each has the three characteristic stages of creep as described by the authors. With a creep-rupture curve of the type shown in Fig. 1 (*a*), the "initiation of tertiary creep," the transition point at 10 per cent increase in creep rate, or the transition point at 100 per cent increase in creep rate will occur at very nearly the same time so that any of these criteria may reasonably be employed as a measure of the useful life.

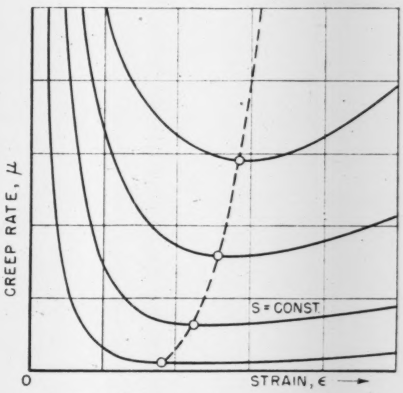

FIG. 2.—Creep Rate *versus* Strain At Constant Stress.

However, with a creep-rupture curve of the type shown in Fig. 1 (*b*), where you have a gradual increase in the creep rate, the useful life of the material may extend well beyond the "initiation of the third stage" or the "transition point at 10 per cent" increase in creep rate over the minimum rate. In some designs employing materials which give this type of curve, we can use the time to reach the "transition point at 100 per cent" increase in creep rate over the minimum rate, as this time may be at about half the rupture life.

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[7] Research Engineer, Mechanics Dept., Research Labs., Westinghouse Electric Corp., East Pittsburgh, Pa.

[8] M. J. Manjoine, "New Machine for Creep and Creep-Rupture Tests" *Transactions*, Am. Soc. Mechanical Engrs., Vol. 67, pp. 111–116 (1945).

We realize the inadequacy of these measurements of the useful life of a material and we hope that in the future this Society might establish a standard which will clearly define the "point" in question.

MR. GOHN.—Would it help to plot the creep rates against time, rather than against strain?

MR. MANJOINE.—There is a big difference between plots of "creep rate *versus* time" and "creep rate *versus* strain." If you use the plot of creep rate *versus* time for low stresses, the transition point is approached at such long times that the extrapolation is difficult. If, however, the creep rate is plotted *versus* strain as in Fig. 2,[9] the minimum creep rate (see dotted curve) and the transition point can readily be determined for each stress level and an extrapolation can be made for these points to curves of lower stresses where the test may have been stopped before the minimum creep rate was reached.

We feel that there should be some standardization as to the per cent increase to be allowed over the minimum rate to define the transition point.

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[9] A. Nadai, "The Influence of Time Upon Creep—The Hyperbolic Sine Creep Law," Stephen Timoshenko Anniversary Volume, pp. 155–170, Macmillan Co., Inc., New York, N. Y. (1938).

CREEP CHARACTERISTICS OF PHOSPHORIZED COPPER (0.019 PER CENT P) AT 300, 400, AND 500 F[*]

By A. I. Blank[1] and H. L. Burghoff[1]

Synopsis

Creep tests at 300, 400, and 500 F were made on phosphorized copper of 0.019 per cent phosphorus content in three annealed and three cold-drawn tempers.

The creep strength of the annealed copper increases with grain size at these temperatures. The relative lowering of creep strength produced by increasing temperature diminishes as grain size increases. The creep strength of the drawn material at 300 F increases with degree of cold working but decreases at 400 and 500 F as softening becomes a factor.

The results are compared with those previously obtained by the authors on other types of copper.

This paper, the fourth in a series of papers (1, 2, 3)[2] dealing with creep characteristics of various types of copper and copper alloys at 300, 400, and 500 F, presents the results of creep tests on phosphorized copper of 0.019 per cent phosphorus content. This copper is designated as DHP, Phosphorized, high residual phosphorus, deoxidized copper in ASTM Specification B 224 - 48 T.[3] One of the previous papers (2) in the series reported the results of similar tests on DLP copper,[3] low residual phosphorus content, 0.008 per cent, in the form of $\frac{1}{8}$-in. diameter wire (*a*) as drawn 84 per cent and (*b*) as drawn 84 per cent and annealed to 0.013 mm average grain size. In the case of the present copper of 0.019 per cent phosphorus content, a more extensive study was made of the effect on creep properties of degree of reduction for hard-drawn specimens and of grain size for annealed specimens.

In 1944 Parker and Riisness (4) reported that the creep properties of annealed oxygen-free high-conductivity copper (OF)[3] at 200 C were independent of grain size. These authors indicated, however, that the creep strength might vary with grain size at other temperatures. The creep properties of metals generally have been found to be influenced by grain size. Hanson (5) and Gillett (6) cited numerous examples in this connection. Results obtained in the earlier investigations by the present authors (1, 3) showed that the creep properties of annealed brasses and other copper alloys are dependent on grain size.

Schwope, Smith, and Jackson (7) determined the effect of various degrees of cold work on the creep properties of tough pitch, oxygen-free, and silver-bearing coppers. Their results showed that the gain in creep strength effected by cold work is lost when softening occurs at elevated temperature. They also reported the effectiveness of silver additions in raising the creep strength of copper.

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[*] Presented at the Fifty-fourth Annual Meeting of the Society, June 18-22, 1951.

[1] Assistant Research Metallurgist and Research Metallurgist, respectively, Chase Brass and Copper Co., Waterbury, Conn.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 992.

[3] Tentative Classification of Coppers (B 244 - 48 T), 1949 Book of ASTM Standards, Part 2, p. 346.

Another recent contribution to the knowledge of the creep of copper was that of Jenkins and Digges (8) which described the effect of thermal and strain history, loading rate, and other factors on the creep of annealed, oxygen-free high-conductivity copper.

MATERIAL

In order to investigate the effects of grain size and degree of reduction on creep properties, the phosphorized copper was prepared in three annealed tempers and three drawn tempers, namely, as annealed to 0.070, 0.032, and 0.015 mm average grain sizes and as drawn 21, 37, and 84 per cent. All of the materials for testing were in the form of $\frac{1}{8}$-in. diameter wire which was processed from two 3-in. diameter cast billets by forging, rolling, and drawing. Both billets had the following chemical composition: 99.98 per cent copper, 0.019 per cent phosphorus, <0.01 per cent zinc, <0.003 per cent iron, <0.002 per cent nickel, <0.001 per cent lead. The three lots of hard-drawn wire were annealed to 0.040 mm average grain size on the ready-to-finish diameters of 0.141, 0.157, and 0.312 in., and were then drawn 21, 37, and 84 per cent, respectively, to 0.125-in. final diameter. The wire was mechanically straightened and cut in 5-ft lengths for use as creep test specimens.

TABLE I.—PHYSICAL PROPERTIES OF MATERIALS TESTED.
Tests at 300, 400, and 500 F were made after exposure of 24 hr at temperature.

	Testing Temperature, deg Fahr	As Annealed			As Drawn		
		0.070 mm Grain Size	0.032 mm Grain Size	0.015 mm Grain Size	21 per cent	37 per cent	84 per cent
Stress at 0.2 per cent Offset, psi	70	3550	4950	7850	38 800	40 000	50 700
	300	3050	4450	7600	34 500	36 600	44 000
	400	3000	4250	6600	29 200	32 600	38 400
	500	2650	3800	6250	24 600	26 900	12 600
Yield Strength at 0.5 per cent. Extension Under Load, psi	70	4550	5850	8600	40 100	41 200	52 000
	300	4000	5250	8300	35 100	37 200	44 300
	400	3900	5050	7300	30 300	33 300	39 200
	500	3500	4500	6950	26 000	28 000	14 300
Tensile Strength, psi	70	32 200	33 300	34 800	42 700	45 000	56 400
Elongation in 2 in., per cent	70	49	49	50	14	12	10
Electrical Conductivity, per cent IACS (International Annealed Copper Standard)	70	88.6	89.0	89.0	88.7	88.7	88.5

The annealed tempers with grain sizes of 0.015 and 0.032 mm were prepared from wire previously annealed to 0.040 mm average grain size and drawn 84 per cent, and that with 0.070 mm final grain size from wire previously annealed to 0.070 mm grain size and drawn only 37 per cent. The annealed specimens in each case were straightened and cut to length prior to the final annealing operation.

Physical properties of the copper in each of the six tempers are given in Table I.

PROCEDURE

The creep-testing equipment and the method of testing are considered only briefly since they have been described in

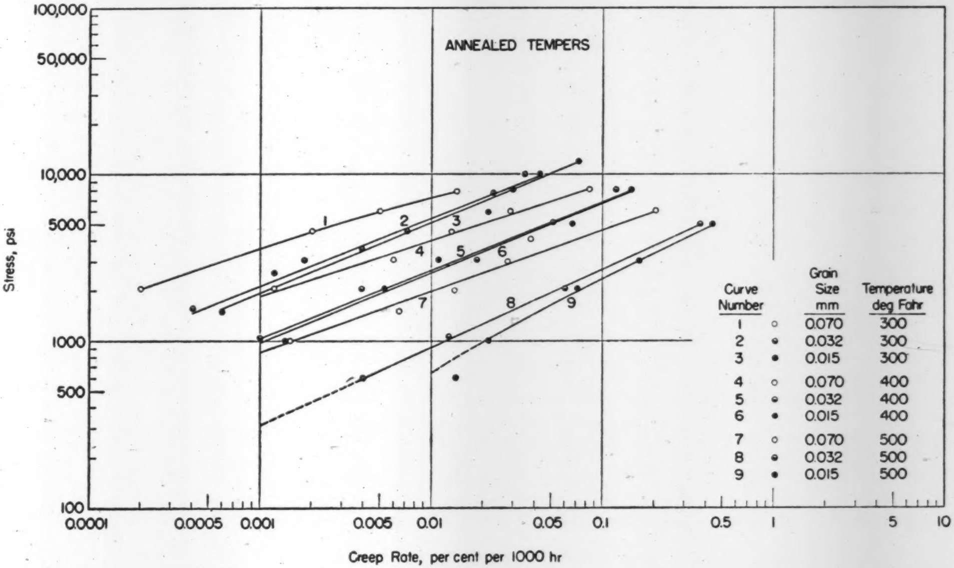
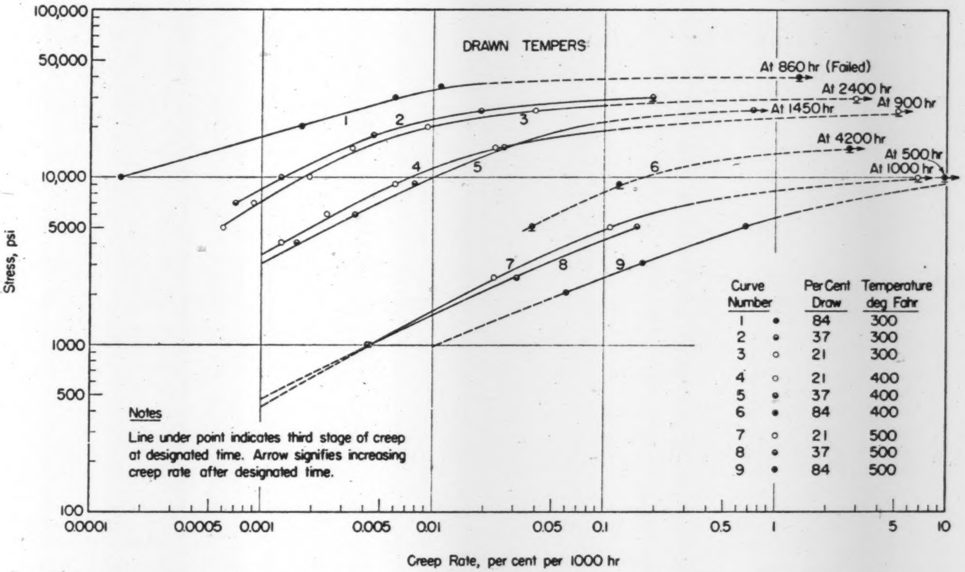


FIG. 1.—Stress and Creep Rate Relationships (Log Scales) at 300, 400, and 500 F for Annealed Phosphorized Copper (0.019 per cent P).



Line under point indicates third stage of creep at designated time. Arrow signifies increasing creep rate after designated time.

FIG. 2.—Stress and Creep Rate Relationships (Log Scales) at 300, 400, and 500 F for Hard-Drawn Phosphorized Copper (0.019 per cent P).

detail previously (1, 2, 3). The strain in the specimens was measured over a 10-in. gage length by means of dial indicators having 0.0001-in. scale divisions. The full range of the indicators provided for a total extension of 2.5 per cent in the 10-in. gage length. When the total extension exceeded 2.5 per cent, as it did in

TABLE II.—RESULTS OF CREEP TESTS.

Temper	Testing Temperature, deg Fahr	Stress, psi	Total Time, hr	Initial Extension, per cent	Total Creep, per cent	Creep Intercept, e_o,[a] per cent	Creep Rate, v,[a] per cent per 1000 hr
Annealed, 0.070 mm grain size	300	2 050	6000	0.045	0.028	0.027	0.0002
		4 550	6000	0.624	0.240	0.228	0.0020
		6 000	6000	1.042	0.378	0.348	0.0050
		7 850	6000	1.772	0.619	0.535	0.014
	400	2 050	6000	0.033	0.036	0.029	0.0012
		3 050	6000	0.185	0.230	0.194	0.0060
		4 500	7080	0.647	0.451	0.359	0.013
		6 000	6000	1.042	0.820	0.647	0.029
		8 000	7080	1.830	1.630	1.040	0.084
	500	1 050	6000	0.012	0.018	0.009	0.0015
		2 000	6000	0.044	0.188	0.106	0.0137
		4 050	6000	0.447	0.960	0.733	0.038
Annealed, 0.032 mm grain size	300	1 550	7080	0.008	0.021	0.018	0.0004
		3 050	6600	0.053	0.043	0.031	0.0018
		3 550	7080	0.085	0.157	0.129	0.0039
		4 550	7200	0.250	0.320	0.268	0.0072
		7 750	6000	1.165	0.620	0.685	0.023
		10 000	7200	2.080	1.360	1.095	0.037
	400	1 050	7080	0.007	0.0125	0.0055	0.0010
		2 050	6000	0.018	0.060	0.037	0.0039
		3 050	7080	0.060	0.295	0.164	0.0185
		5 080	7080	0.358	1.020	0.660	0.051
		8 050	6000	1.500	1.840	1.120	0.120
	500	600	6900	0.006	0.042	0.015	0.0040
		1 050	6000	0.010	0.090	0.014	0.0127
		2 050	6000	0.025	0.467	0.104	0.0605
		5 000	6000	0.603	3.577	1.327	0.375
Annealed, 0.015 mm grain size	300	1 500	7080	0.011	0.027	0.023	0.0006
		2 550	7080	0.016	0.035	0.0265	0.0012
		5 950	6000	0.079	0.279	0.151	0.021
		8 050	6000	0.233	1.155	0.973	0.030
		10 000	6000	1.015	1.465	1.205	0.043
		11 900	6000	1.575	1.825	1.395	0.072
	400	1 000	7080	0.007	0.023	0.013	0.0014
		2 050	6000	0.013	0.055	0.023	0.0053
		3 050	6000	0.026	0.121	0.054	0.011
		5 000	7080	0.075	0.800	0.325	0.067
		8 000	6000	0.560	2.530	1.640	0.148
	500	600	6900	0.006	0.114	0.016	0.0143
		1 000	6900	0.008	0.157	0.009	0.0215
		2 050	6000	0.014	0.460	0.031	0.0715
		3 000	6000	0.025	1.283	0.305	0.163
		5 000	6900	0.058	4.640	1.600	0.44
		6 850	2400	0.467	5.828	2.178	1.52
Drawn 21 per cent	300	5 000	7080	0.035	0.021	0.0165	0.0006
		7 000	6000	0.050	0.028	0.023	0.0009
		10 000	6000	0.070	0.064	0.053	0.0019
		14 950	6000	0.105	0.087	0.066	0.0034
		20 000	6000	0.139	0.177	0.122	0.0092
		25 000	6000	0.182	0.427	0.191	0.039
		29 900	2400	0.210	4.090	−3.000	2.96[b]
	400	4 050	7080	0.028	0.037	0.0275	0.0013
		6 000	7080	0.043	0.056	0.039	0.0024
		9 100	7080	0.062	0.0935	0.051	0.006
		15 000	6000	0.112	0.288	0.148	0.023
		25 000	900	0.237	4.175	−0.485	5.18[b]
	500	1 000	6900	0.007	0.071	0.041	0.0043
		2 500	6900	0.018	0.516	0.358	0.023
		5 000	6000	0.036	2.064	1.417	0.108
		10 000	1000	0.075	3.855	−2.900	6.755[b]
		20 350	290	0.206	5.22	—	[b]

TABLE II.—*Continued*

Temper	Testing Temperature, deg Fahr	Stress, psi	Total Time, hr	Initial Extension, per cent	Total Creep, per cent	Creep Intercept, e_o,[a] per cent	Creep Rate, v,[a] per cent per 1000 hr
Drawn 37 per cent	300	7 000	7080	0.048	0.020	0.015	0.0007
		10 000	6000	0.068	0.052	0.044	0.0013
		17 950	6000	0.123	0.127	0.100	0.0045
		25 000	6000	0.180	0.289	0.177	0.019
		30 100	5300	0.216	1.344	0.364	0.185^{c}
		30 100	6000	0.216	1.478	0.325	0.192^{b}
		34 900	340	0.298	3.593^{d}	...	...
	400	4 050	7080	0.028	0.035	0.024	0.0016
		6 000	7080	0.041	0.059	0.034	0.0035
		9 100	7080	0.062	0.129	0.074	0.0078
		15 000	6000	0.104	0.304	0.147	0.026
		25 150	1450	0.196	1.390	0.324	0.735^{c}
		25 150	6000	0.196	7.080	−5.760	2.14^{b}
		30 000	240	0.235	2.655	...	^{b}
	500	1 000	6900	0.008	0.061	0.032	0.0042
		2 500	6900	0.020	0.552	0.338	0.031
		5 050	6000	0.040	2.460	1.530	0.155
		12 000	220	0.100	3.505	...	^{b}
Drawn 84 per cent	300	10 000	6000	0.065	0.024	0.023	0.00015
		20 000	6000	0.130	0.057	0.047	0.0017
		30 000	6000	0.203	0.170	0.134	0.0060
		34 950	6000	0.245	0.225	0.159	0.011
		39 950	860	0.343	1.197	0.027	1.360^{e}
	400	5 050	7080	0.033	0.186	−0.085	0.038^{b}
		9 050	6000	0.059	0.288	−0.437	0.121^{b}
		9 050	7080	0.059	0.475	−1.110	0.224^{b}
		15 000	580	0.103	0.066	0.034	0.055^{c}
		15 000	4200	0.103	2.710	−8.630	2.70^{b}
		30 000	740	0.215	3.705	...	^{b}
	500	2 050	6000	0.019	0.485	0.124	0.060
		3 050	6000	0.029	1.321	0.306	0.169
		5 100	6000	0.053	5.297	1.277	0.670
		10 000	500	0.160	7.45	2.470	9.96^{b}

[a] The values e_o and v may be used in the McVetty type of formula (9) for calculating amount of creep as follows:

$$\text{Creep} = e_o + vt$$

where:
e_o = a constant (intercept for zero time on creep-time plot),
v = creep rate (slope or tangent to creep curve), and
t = time

[b] Third stage of creep, accelerating creep rate.

[c] This value applies to second stage of creep at indicated time, after which the test entered the third stage of creep.

[d] Total measured creep prior to failure.

[e] Third stage of creep followed by failure shortly after indicated duration of test.

a few instances, the indicators were reset without interrupting the creep tests. Temperatures within the gage length of all specimens were held within the limits specified in ASTM Standard Recommended Practice for Conducting Long-Time High-Temperature Tension Tests of Metallic Materials (E 22 – 41).[4]

The creep tests were run at constant load for periods of time up to 6000 hr or more in most cases. The indicated extensions were recorded at least every two or three days throughout the duration of the tests. When the tests were terminated, the specimens were removed from the furnaces and the portions of the specimens within the gage length were subjected to tension tests at room temperature to determine the effect of the creep-test exposures on the initial physical properties. The effect of exposure on microstructure also was ascertained.

DISCUSSION OF RESULTS OF CREEP TESTS

The data listed in Table II were derived from the basic creep curves, extension *versus* time, and the creep rates

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[4] 1949 Book of ASTM Standards, Part 1, p. 1281.

TABLE III.—SUMMARY OF CREEP PROPERTIES.[a]

Temper	Temperature, deg Fahr	Stress, psi, for Designated Creep Rate		
		0.001 per cent per 1000 hr	0.01 per cent per 1000 hr	0.1 per cent per 1000 hr
Annealed, 0.070 mm grain size	300	3 600	7 200	(>10 000)
	400	1 850	4 000	8 700
	500	850	2 000	4 600
Annealed, 0.032 mm grain size	300	2 100	5 400	(>10 000)
	400	1 000	2 650	6 800
	500	(300)	900	2 700
Annealed, 0.015 mm grain size	300	1 950	5 100	(>12 000)
	400	1 000	2 550	6 700
	500	...	650	2 350
Drawn 21 per cent	300	7 200	20 000	27 000
	400	3 400	11 300	19 000
	500	(400)	1 600	4 850
Drawn 37 per cent	300	8 500	22 100	29 000
	400	3 000	10 000	21 300
	500	(450)	1 500	4 200
Drawn 84 per cent	300	17 300	33 500	(39 000)
	400	...	(2 500)	8 200[b]
	500	...	(1 000)	2 500

[a] Values enclosed in parentheses were derived by extrapolation.

[b] Produces third stage of creep, accelerating creep rate.

in effect at approximately 6000 hr were used as the primary criterion of creep properties. Since the creep rate in most of the tests changed throughout the entire duration of the tests, it was necessary to measure the creep rate at substantially the same time in all tests in order to insure a common basis of comparison. Unless otherwise indicated, the measured creep rates pertain to the first or second stages of creep. In most cases the creep rates were still decreasing very slightly at 6000 hr. Although this implies the first stage of creep by strict definition, such creep curves may be considered for practical purposes to have attained the second stage of creep since the continuing change in creep rate was very small.

The creep rates at 6000 hr were used in plotting the log stress–log creep rate relationships shown in Figs. 1 and 2. For the annealed tempers, these relationships

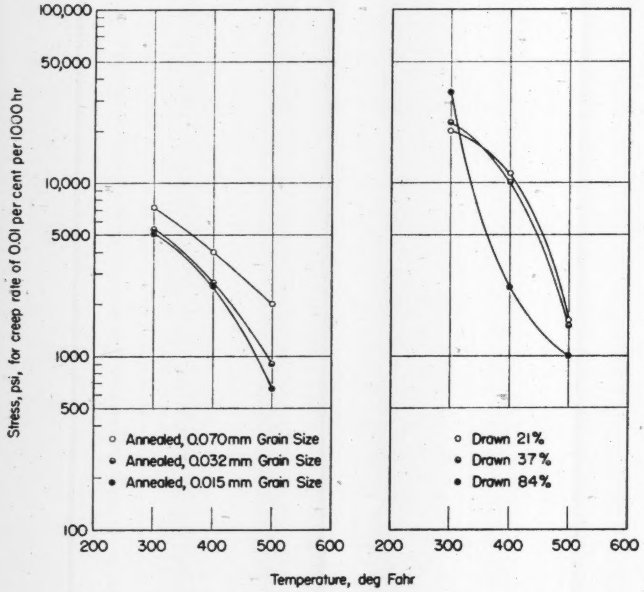

FIG. 3.—Creep Strength (Log Scale) and Temperature Relationships for Annealed and Hard-Drawn Phosphorized Copper (0.019 per cent P).

conform to relatively straight lines of substantially similar slope. Some of the lines have a small amount of curvature which is convex in the direction of increasing stress. In general, the log stress -log creep rate curves for the hard-drawn tempers are also more or less parallel to each other over most of their length, although they tend to converge at higher creep rates. They are relatively straight in the range of creep rates less than 0.05 per cent per 1000 hr and are curved at higher creep rates. The creep properties shown in Figs. 1 and 2 are summarized in Table III. The stresses which produce creep rates of 0.001, 0.01, and 0.1 per cent per 1000 hr at 6000 hr at 300, 400, and 500 F are given in this table.

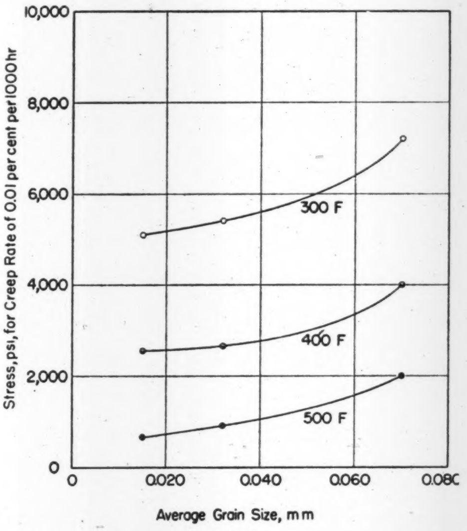

FIG. 4.—Relationships Between Creep Strength and Grain Size for Annealed Phosphorized Copper (0.019 per cent P) at 300, 400, and 500 F.

The values of stress which produce a creep rate of 0.01 per cent per 1000 hr provide a convenient basis of comparison of relative creep strength. These values are shown as functions of temperature in Fig. 3. Creep strength for both the annealed and hard-drawn tempers falls off appreciably as temperature increases from 300 to 500 F. The loss is relatively greater in the case of the drawn tempers. The proportionate decrease in creep strength of the annealed materials due to increasing temperature is less as the grain size increases, although the absolute change in the values of creep strength from 300 to 500 F is substantially the same for all three annealed materials. Creep strength at the indicated temperatures increases with increasing grain size, although the difference in creep strength for 0.015- and 0.032-mm grain sizes is slight. The effect of grain size on creep strength is shown somewhat more effectively in Fig. 4 where creep strength is plotted as a function of grain size.

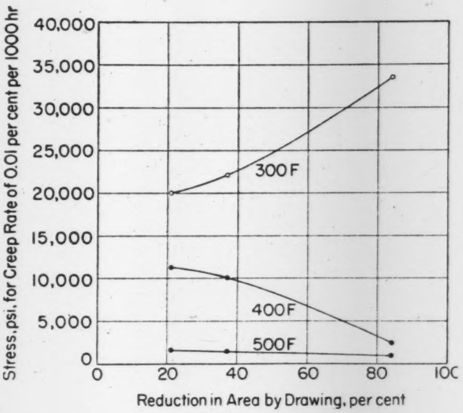

FIG. 5.—Relationships Between Creep Strength and Degree of Reduction for Hard-Drawn Phosphorized Copper (0.019 per cent P) at 300, 400, and 500 F.

Creep strength of the drawn tempers is shown as a function of degree of reduction in Fig. 5. The effect of increasing reduction on creep strength at 300 F is similar to its effect on tensile strength at

TABLE IV.—EFFECT OF CREEP TEST EXPOSURES ON ROOM TEMPERATURE PROPERTIES.

Temper	Creep Testing			Tensile Strength, psi	Elongation in 2 in., per cent	Microstructure
	Temperature, deg Fahr	Stress, psi	Time, hr			
Drawn 21 per cent	(As processed)			42 700	14.0	Hard drawn
	300	0	7200	42 300	17.0	Unchanged
		5 000	7080	42 300	17.0	Unchanged
		10 000	7200	42 250	17.0	Unchanged
		20 000	7200	42 000	17.0	Unchanged
		29 900[a]	1660	42 350	14.5	Unchanged
	400	0	6300	40 000	21.5	Unchanged
		4 050	7080	39 800	23.0	Unchanged
		9 100	7080	39 700	22.5	Unchanged
		15 000	6300	40 000	21.5	Unchanged
		25 000[a]	1000	39 700	26.0	Unchanged
	500	0	240	38 700	25.0	Unchanged
		0	1000	32 800	43.5	Completely recrystallized
		0	8250	31 650	42.5	Completely recrystallized
		1 000	6910	31 800	46.5	Completely recrystallized
		5 000	7200	31 750	39.5	Completely recrystallized
		10 000[a]	1080	32 300	38.0	Completely recrystallized
		20 350[a]	500	34 250	30.5	Partially recrystallized
Drawn 37 per cent	(As processed)			45 000	12.0	Hard drawn
	300	0	7200	45 400	14.5	Unchanged
		10 000	7200	45 400	14.5	Unchanged
		25 000	7200	44 250	15.5	Unchanged
		30 100[a]	7200	43 750	12.0	Unchanged
		34 900[a]	360	45 550	11.0	Unchanged
	400	0	6300	42 300	21.0	Unchanged
		6 000	7080	42 350	20.0	Unchanged
		15 000	6300	42 250	21.0	Unchanged
		25 150[a]	7200	40 950	21.0	Unchanged
		30 000[a]	335	42 850	15.0	Unchanged
	500	0	24	42 600	19.0	Unchanged
		0	240	34 700	41.0	Completely recrystallized
		0	8000	32 150	43.5	Completely recrystallized
		2 500	6910	32 100	49.0	Completely recrystallized
		5 050	7200	31 800	40.0	Completely recrystallized
		12 000[a]	240	32 850	41.0	Completely recrystallized
Drawn 84 per cent	(As processed)			56 400	10.0	Hard drawn
	300	0	7200	54 200	12.5	Unchanged
		10 000	7200	54 350	13.5	Unchanged
		20 000	7200	53 950	11.5	Unchanged
		30 000	7200	53 750	13.0	Unchanged
		39 950[a]	930	52 300	11.0	Unchanged
	400	0	240	53 200	15.5	Unchanged
		0	1000	50 100	18.5	Partially recrystallized
		0	2400	43 100	27.0	Partially recrystallized
		0	5000	37 800	35.0	Completely recrystallized
		5 050	7080	36 800	40.5	Completely recrystallized
		9 050	7080	37 500	40.0	Completely recrystallized
		15 000[a]	4950	37 850	35.0	Completely recrystallized
		30 000[a]	840	45 700	15.5	Partially recrystallized
	500	0	24	37 800	41.0	Partially recrystallized
		0	240	35 400	46.0	Completely recrystallized
		0	7420	33 850	44.0	Completely recrystallized
		2 050	7200	34 200	42.0	Completely recrystallized
		5 100	8180	34 400	42.0	Completely recrystallized
		10 000[a]	530	35 850	39.5	Completely recrystallized

[a] Produced third stage of creep, accelerating creep rate.

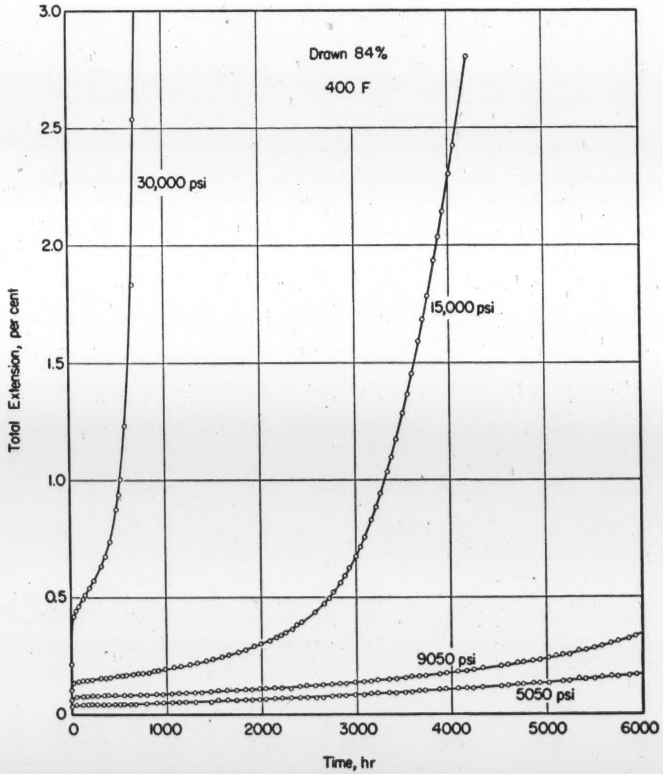

FIG. 6.—Time-Extension Curves at 400 F for Hard-Drawn Phosphorized Copper (0.019 per cent P), 84 per cent Reduction.

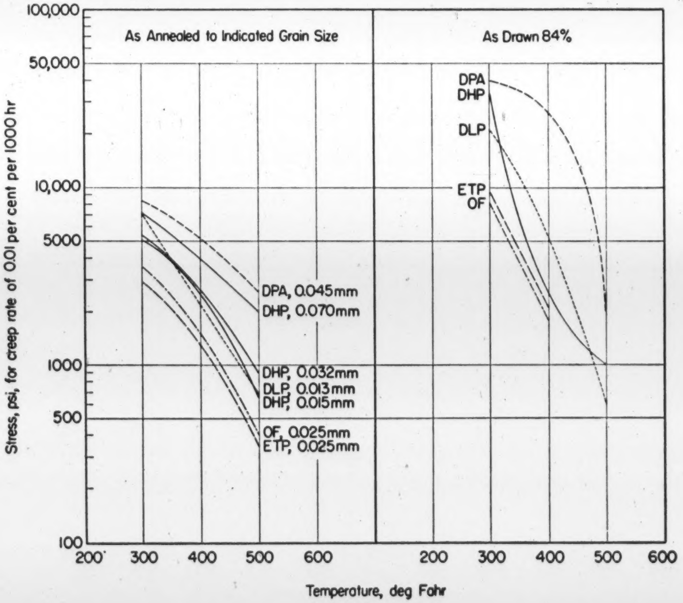

FIG. 7.—Creep Strength (Log Scale) and Temperature Relationships for Some Annealed and Hard-Drawn (84 per cent Reduction) Coppers.

TABLE V.—COMPARISON OF SEVERAL TYPES OF COPPER WITH RESPECT TO TENSILE PROPERTIES AND ELECTRICAL CONDUCTIVITY AT 70 F AND CREEP STRENGTH AT 300, 400 AND 500 F.

ASTM Designation	Reference	Temper		Tensile Strength at 70 F, psi	Yield Strength at 0.5 per cent Extension Under Load at 70 F, psi	Creep Strength, Stress, psi, for Creep Rate of 0.01 per cent per 1000 hr			Electrical Conductivity at 70 F, per cent IACS
		Annealed Average Grain Size, mm	Drawn Reduction, per cent			300 F	400 F	500 F	
ETP	(3)	0.025	..	35 100	7 200	2 950	1 300	350	100.9
OF	(3)	0.025	..	34 500	7 900	3 600	1 500	400	101.3
DLP	(2)	0.013	..	36 500	12 000	7 200	2 100	700	100.1
DHP		0.015	..	34 800	8 600	5 100	2 550	650	89.0
		0.032	..	33 300	5 850	5 400	2 650	900	89.0
		0.070	..	32 200	4 550	7 200	4 000	2000	88.6
DPA	(3)	0.045	..	35 000	6 100	8 400	5 200	2600	45.3
ETP	(3)	...	84	55 400	50 000	9 400	2 100	...	99.1
OF	(3)	...	84	54 500	49 500	8 300	1 750	...	99.5
DLP	(2)	...	84	57 500	54 000	21 000	5 200	600	98.3
DHP		...	84	56 400	52 000	33 500	2 500[a]	1000[a]	88.5
DPA	(3)	...	84	60 900	55 500	40 000	27 500	2000[b]	46.0

[a] By extrapolation of log stress-log creep rate curve.
[b] Produces third stage of creep, accelerating creep rate.

room temperature, since both increase with degree of cold working. At 400 and 500 F, however, the trend is reversed and the more severely worked material has lower creep strength, the effect being associated with softening during the creep tests. The relative stabilities of the three hard-drawn materials at the test temperatures are indicated by the data in Table IV in which tensile strength and elongation at room temperature before and after various creep test exposures are listed. At 400 F, the material drawn 84 per cent recrystallized and softened during the course of the creep tests, whereas the two less severely drawn tempers were relatively stable at this temperature. All three drawn tempers recrystallized relatively early in the creep tests at 500 F, and, once softening occurred, the deformation was characteristic of annealed material. The average grain sizes of the materials initially drawn 21, 37, and 84 per cent were approximately 0.070, 0.050, and 0.025 mm, respectively, at the end of the creep tests at 500 F. When compared with the creep strength of the initially annealed tempers at 500 F, the creep strength of the initially drawn tempers at 500 F is found to be remarkably consistent with the grain sizes attained during the creep tests at this temperature, even though the grain sizes produced by softening in the creep tests were very non-uniform because of the relatively low rates of recrystallization.

TABLE VI.—IDENTIFICATION OF COPPERS.

ASTM Designation	Type of Copper	Composition of Material Tested
ETP	Electrolytic tough pitch	99.96 Cu, <0.002 Fe, <0.002 Ni, <0.001 Pb, <0.0005 Sn
OF	Oxygen-free without residual deoxidants	99.98 Cu, <0.01 Zn, <0.003 Fe, <0.005 Ni, <0.0005 Pb
DLP	Phosphorized, low residual phosphorus	99.95 Cu, 0.008 P, 0.001 Fe, 0.001 Ni
DHP	Phosphorized, high residual phosphorus	99.98 Cu, 0.019 P, <0.01 Zn, <0.003 Fe, <0.002 Ni, <0.001 Pb
DPA	Phosphorized, arsenical	99.60 Cu, 0.32 As, 0.022 P, 0.015 Fe, 0.005 Ni, 0.0005 Pb, <0.01 Zn, <0.002 Sb, <0.001 Mn

The gradual recrystallization of the material of 84 per cent reduction during a major portion of the creep test period at 400 F markedly affected the rate of deformation. This is evident in the basic creep curves shown in Fig. 6. The tests at the two higher applied stresses quickly progressed to the third stage of creep. Similarly, but much more slowly, the creep rate in the tests at the two lower stresses started to increase after about 500 hr. Considering the extremely slow increase in creep rate in these two tests, this may not have been the final stage of creep. It is conceivable that the creep rate in these tests might have stabilized after a much longer time of exposure. Behavior of this type has previously been observed (1) in other cases where recrystallization occurred during a portion or all of the creep test exposure, for example, cartridge brass, drawn 37 per cent, at 400 F.

In some of the exposures on the drawn wire, a thin layer at the surface recrystallized more rapidly than the major portion of the cross-section. This was found to be a consequence of internal oxidation during one or more of the anneals in processing the wire. It was not due, as stated in one of the previous papers in this series (2), to working of the surface layers of the wire in the straightening operation. Instead, the removal of phosphorus from solid solution by internal oxidation made the affected layer more susceptible to softening. However, the extent of this condition was sufficiently slight as to have no appreciable effect on the creep test results.

The creep strength of this phosphorized copper of 0.019 per cent phosphorus content is compared in Fig. 7 with the creep strength of several other types of copper in corresponding tempers. The data for the latter were taken from two previously published papers (2, 3). All of these coppers were likewise tested in the form of $\frac{1}{8}$-in. diameter wire. The stresses which produced a creep rate of 0.01 per cent per 1000 hr at 6000 hr are listed in Table V for ETP, OF, DLP, DHP and DPA coppers (See Table VI for chemical composition),[3] as annealed to various grain sizes and as drawn 84 per cent. These stresses for the phosphorized coppers, DLP and DHP, are between those for ETP and OF coppers on the one hand and those for arsenical copper, DPA, on the other hand, at 300, 400, and 500 F.

The relative order of creep strength of the two phosphorized coppers which were tested, DLP and DHP, changes for different conditions and different temperatures. As annealed to 0.013 mm (DLP) and 0.015 mm (DHP) grain sizes, these coppers have approximately equal creep strength at 400 and 500 F, but at 300 F the DLP copper appears to be significantly superior to the DHP copper. The stresses which produced the creep rate of 0.01 per cent per 1000 hr at 6000 hr are 7200 and 5100 psi, respectively, for the DLP and DHP coppers at 300 F. This difference in favor of the copper of lower phosphorus content is considered to be associated with its appreciably higher yield strength at room temperature and at moderately elevated temperatures such as 300 F; it is not considered to be an inherent characteristic.

The values of yield strength at 0.5 per cent extension under load at 70 and 300 F were 8600 and 8300 psi, respectively, for the DHP copper, 0.015-mm grain size, whereas the corresponding values for the DLP copper, 0.013-mm grain size, were 12,200 and 10,800 psi, respectively. The higher values of yield strength for the DLP copper may be attributed to the fact that its ready-to-finish grain size was only about 0.020 mm, whereas that of the DHP copper was 0.040 mm. This difference may have imparted a greater degree of preferredness in the DLP copper which would account for its higher yield strength. The conditions which produced the higher yield strength in the annealed DLP copper were in all probability responsible also for its greater creep strength at 300 F in comparison with that of DHP copper, 0.015-mm grain size.

For the condition as drawn 84 per cent,

the DHP copper had greater creep strength than the DLP copper at 300 and 500 F. The exception at 400 F, where the DHP copper had lower creep strength than the DLP copper, is most likely associated with differences in their softening characteristics at this temperature. The prolonged structural change in the DHP copper, drawn 84 per cent, at 400 F would account at least in part for its relatively low creep strength at this temperature.

Since the use of copper in some applications depends on electrical conductivity and creep strength, both of these properties are listed in Table V for the various coppers under consideration. It is evident from these data that the phosphorized copper of low phosphorus content, DLP, combines high electrical conductivity with intermediate creep strength, in relation to the corresponding properties for the other types of copper. The phosphorus content in DLP copper is not large enough to have much effect on electrical conductivity, but it is sufficient to increase the resistance to softening and creep. In this connection silver-bearing copper might also be expected to have relatively high creep strength in combination with its high electrical conductivity, since the silver content is known to impart substantially increased resistance to softening.

SUMMARY

Creep characteristics of phosphorized copper (DHP) of 0.019 per cent phosphorus content were determined at 300, 400, and 500 F. The material was tested in the form of $\frac{1}{8}$-in. diameter wire as annealed to grain sizes of 0.015, 0.032, and 0.070 mm, and as drawn 21, 37, and 84 per cent in order to investigate the effects of grain size and degree of reduction on its creep properties.

In the annealed condition, creep strength increases with grain size at all three temperatures. The decrease in creep strength as temperature increases is relatively less for larger grain size. For the drawn tempers, creep strength increases with reduction at 300 F and decreases with increasing reduction at 400 and 500 F because of softening at the two higher temperatures.

The creep strength of the DHP copper is greater than that of the ETP and the OF coppers and less than that of the DPA copper which have been previously tested by the authors. Except for relatively minor differences due to slight differences in actual temper in the case of the materials tested, the DHP and DLP coppers have generally similar creep properties.

Acknowledgment:

The authors acknowledge the interest of D. K. Crampton, Director of Research of the Chase Brass and Copper Co., in the creep testing program which has been in progress for a number of years in the research laboratories of the company. The assistance of those who participated in various phases of the work leading to this paper is acknowledged. Permission to publish the results was given by the Chase Brass and Copper Co.

REFERENCES

(1) H. L. Burghoff, A. I. Blank and S. E. Maddigan, "The Creep Characteristics of Some Copper Alloys at Elevated Temperatures," *Proceedings*, Am. Soc. Testing Mats., Vol. 42, p. 668 (1942).

(2) H. L. Burghoff and A. I. Blank, "Creep Characteristics of A Phosphorized Copper," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 161, p. 420 (1945).

(3) H. L. Burghoff and A. I. Blank, "The Creep Characteristics of Copper and Some Copper Alloys at 300, 400 and 500 F," *Proceedings*, Am. Soc. Testing Mats., Vol. 47, p. 725 (1947).

(4) E. R. Parker and C. F. Riisness, "Effect of Grain Size and Bar Diameter on Creep Rate of Copper at 200 C," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 156, p. 117 (1944).

(5) D. Hanson, "The Creep of Metals," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 133, p. 15 (1939).

(6) H. W. Gillett, "Some Things We Don't Know About The Creep of Metals," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 135 p. 15 (1939).

(7) A. D. Schwope, K. F. Smith and L. R. Jackson, "The Comparative Creep Properties of Several Types of Commercial Coppers," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 185, p. 409 (1949).

(8) W. D. Jenkins and T. G. Digges, "Creep of High-Purity Copper," *Journal of Research*, Nat. Bureau Standards, Vol. 45, No. 2, p. 153 (1950).

(9) P. G. McVetty, "The Interpretation of Creep Tests," *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part II, p. 105 (1934).

DISCUSSION

MR. W. A. REICH.[1]—The authors are to be congratulated on presenting these data, since interest in high-temperature properties continues apace in the design of electrical equipment which is expected, of course, to operate at higher and higher temperatures as time goes on.

Referring to Fig. 1 of the paper, in speculating on the effect of grain size on the creep properties of materials, one must take into account the mechanism of creep.

It is postulated that at low stresses the mechanism of creep tends toward that of flow at the grain boundaries, and therefore one should expect that at low stresses and low creep rates the effect of grain size would be greatest, and these curves seem to indicate that very nicely.

The slope of curve 1 is much less than that of curves 2 and 3; and the same is true of 4 compared with 5 and 6, and with 7 compared with 8 and 9.

The curves for each temperature diverge most at low creep rates where "grain boundary rotation" is the most likely mechanism of creep.

The authors' comments on this would be appreciated.

MR. J. T. RICHARDS[2] (*by letter*).—On the basis of creep tests conducted on phosphorized copper at 300, 400, and 500 F, have the authors any data regarding the following related points?

1. Creep characteristics of phosphorized copper at room temperature.
2. Influence of grain size and cold drawing on the tension modulus of the material tested.
3. Effect of creep test exposure on room temperature tension modulus and electrical conductivity.
4. Tension modulus at exposure temperature, considering the possible influence of time.
5. Rate of recovery as compared to creep rate.

The authors among others have been instrumental in making available much needed information on the anelastic characteristics (creep, relaxation, etc.) of copper and its alloys; however, little has been published regarding the influence of temperature on elastic properties. Creep data are helpful in determining design stresses, but there is also a need for information covering the relationship between stress and strain within the limits established by creep tests.

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[2] Development Engineer, The Beryllium Corp., Reading, Pa.

Although it has been known for some time that increases in temperature cause a decrease in elastic modulus, the influence of time at temperature has apparently not been determined. Since overstrain has considerable bearing on elasticity, it is probable that the plastic deformation resulting from creep will exert a similar influence. Creep tests conducted by Laurent and Eudier[3] on an aluminum-copper alloy at room temperature indicate that creep (at least to 64 hr) causes a decrease in modulus similar to tensile overstrain.

In view of the authors' extensive creep testing experience, it is hoped that they will be able to clear up some of these questions.

MESSRS. E. J. DULIS[4] AND G. V. SMITH[4] (*by letter*).—In Table II, footnote *a* states that reported e_0 and v values may be used in the McVetty type of formula Creep $= e_0 + vt$, for calculating total creep. This is true only when the following conditions prevail:

e_0 = intercept of the minimum creep rate extrapolated to zero time axis,
v = minimum or "constant" creep rate, and
t = time up to beginning of third stage of creep.

It is clear, then, that e_0 will be too small when a tangent is drawn from either the first (decelerating) or third (accelerating) stage of creep. Moreover, the quantity v defines one point on a curve when it is not the constant (minimum) creep rate and it cannot be used in the equation unless it is the constant creep rate. Therefore, the e_0 and v values in Table II distinguished by the superscript *b*, indicating third stage of creep, are not applicable to the formula for calculating total creep and may introduce considerable error. Also the e_0 for the 6000-hr tests that had not reached a constant creep rate are in error by an amount which depends on how closely the constant creep rate had been approached.

MR. A. I. BLANK (*author*).—Mr. Reich's observation offers a most likely explanation for the differences in slope of the log stress–log creep rate curves for the annealed materials. Creep at low strain rates has been shown to proceed primarily by movement or diffusion at the grain boundaries. Under these conditions the resistance to creep should be greater for material of larger grain size since there is relatively less grain surface available.

We have relatively few data available to answer the interesting questions raised by Mr. Richards. To date we have not had the opportunity to determine the creep properties of this phosphorized copper at room temperature. Neither have we measured tension modulus or electrical conductivity after exposure in the creep tests at the elevated temperatures. Presumably Mr. Richards refers to recovery in the sense of stress relief and recrystallization of the hard-drawn materials when exposed at the elevated temperatures and not to strain recovery which occurs when the applied stress is removed at the end of the creep test. The data in Table IV provide some measure of the stability of the hard-drawn materials for various exposures.

We have determined stress-strain relationships for the annealed and hard-drawn materials at 70, 300, 400 and 500 F, but because the proportional limit of copper, particularly as annealed, is exceedingly low, it was not possible to derive precise values of modulus of elasticity from the stress-strain curves. A slight decrease in slope of the stress-strain curves with increasing temperature was observed.

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[3] Pierre Laurent and Michel Eudier, "The Influence of Plastic Deformation on the Modulus of Elasticity," *Comptes rendus*, Vol. 228, pp. 225–226 (1949).

[4] Research Laboratory, United States Steel Co., Kearny, N. J.

Values of Young's modulus of elasticity for silver-bearing and silver-free Tough Pitch and OFHC coppers at elevated temperatures were recently reported[5] by Benson, McKeown, and Mends as follows:

Temperature	Young's Modulus of Elasticity, psi
130 C (266 F)	16 000 000
175 C (347 F)	15 500 000
225 C (437 F)	15 000 000

Replying to the discussion by Dulis and Smith, the McVetty type formula for calculating total creep obviously does not describe the creep curve since it is an expression which defines a straight line only. Few, if any, creep curves for copper or the common copper alloys at elevated temperatures attain a constant creep rate within the duration of most creep tests—less than 10,000 hr. Of necessity, therefore, the slope of the basic creep curve, extension *versus* time, at any given time represents the creep rate at that time only. The McVetty type formula (creep= $e_0 + vt$) defines the straight line which is tangent to the creep curve at the time, t, under consideration and which intercepts the ordinate at the strain intercept, e_0. This simple and convenient method of evaluating creep characteristics is quite widely used and is quite adequate for numerous design applications. Except for those instances where the change in creep rate with time is appreciable, the error resulting from the use of this formula is relatively small.

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[5] N. D. Benson, J. McKeown and D. N. Mends, "The Creep and Softening Properties of Copper for Alternator Rotor Windings," *Journal*, Inst. Metals, Vol. 80, Part 3, Nov., 1951, p. 131.

MECHANICAL PROPERTIES OF COPPER AT VARIOUS TEMPERATURES*

By W. H. Munse[1] and N. A. Weil[1]

Synopsis

This paper presents the results of a number of copper coupon tests conducted at temperatures ranging from −321 F to 400 F. Values of yield strength, ultimate strength, reduction of area, elongation in 2 in., and modulus of elasticity were determined for the coppers at each of several temperatures.

Three types of base material have been included in these studies: electrolytic tough-pitch copper, deoxidized high-phosphorus copper, and oxygen-free high-conductivity copper. These three materials have been tested in two thicknesses (⅛ in. and ¼ in.) and in two tempers (annealed and cold-rolled 5 to 7 per cent).

A supplementary series of tests was conducted on the cold-rolled deoxidized high-phosphorus copper at temperatures ranging from −321 F to 1200 F. This series was run to give a more complete picture of the effect of temperature on the properties of this one material.

Another phase of this paper is concerned with a correlation of the results of these tests with the results of tests reported in the literature for the same and similar materials. This correlation provides an extensive study of the effect of temperature on the mechanical properties of various coppers.

In recent years it has become apparent that there is a need for more information on the mechanical properties of coppers at various temperatures. This need has been brought about, to a large extent, by the development of many new and varied structures for which copper is well suited and by the great variety of conditions to which these strructures are subjected. In an attempt to provide some of the desired information, the Copper and Brass Research Assn. initiated an investigation to obtain experimental data suitable for use in the design of copper structures.

This paper deals only with the first phase of the investigation, namely, the mechanical properties of copper coupons at various temperatures. Three types of base materials were included in the studies: electrolytic tough-pitch copper, deoxidized high-phosphorus copper, and oxygen-free high-conductivity copper. These three types of copper were tested in two tempers: annealed and cold-rolled (5 to 7 per cent), and in two thicknesses: $\frac{1}{8}$ in. and $\frac{1}{4}$ in. Each was hot rolled and then annealed at 1150 F for one-half hour. One half was shipped to the laboratory in this condition, whereas the other half was cold rolled.

Another variable included in the program was the orientation of the direction of rolling with respect to the longitudinal axis of the test specimen; it was either parallel or normal. The tests, therefore, included three coppers in two tempers, two thicknesses, two directions of rolling, and six testing temperatures ranging from −321 F to 400 F.

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* Presented at the Fifty-fourth Annual Meeting of the Society, June 18–22, 1951.

[1] Research Assistant Professor of Civil Engineering, and Research Assistant in Civil Engineering, respectively, University of Illinois, Urbana, Ill.

An additional series of tests was conducted on the deoxidized high-phosphorus cold-rolled copper at a number of temperatures between −321 and 1200 F.

The mechanical properties studied in this investigation include the following:

(*a*) Yield point as determined at a 0.2 per cent offset.
(*b*) Yield point as determined at a 0.5 per cent elongation, under load.
(*c*) Ultimate tensile strength.
(*d*) Elongation in 2 in.
(*e*) Reduction of area.
(*f*) Initial slope of the stress-strain curve (modulus of elasticity).

In view of the large number of variables and properties studied and the number of tests conducted, only a brief description of the materials, testing equipment and testing procedure is included in this paper.

Description of Tests:

The test specimens for this investigation were standard rectangular tension specimens with a 2-in. gage length, similar to Fig. 2 of ASTM Standard Methods of Tension Testing of Metallic Materials (E 8 - 46).[2] The tests were performed in a 30,000-lb Riehle Universal Testing Machine or a 120,000-lb Baldwin-Southwark Hydraulic Universal Testing Machine. During the tests, these machines were operated continuously to insure uniform test conditions and to eliminate, insofar as possible, variations from test to test due to creep. The specimens were tested in duplicate at six different temperatures: −321, −90, −40, room temperature (approximately 75 F), 212, and 400 F. An additional series of tests was performed on the $\frac{1}{4}$-in. cold-rolled deoxidized high-phosphorus copper at temperatures of −230, −160, 0, 140, 300, 550, 700, 850, 920, 1000, 1100, and 1200 F.

A second supplementary series of tests was conducted to obtain information concerning the effect of the speed of loading on the mechanical properties of one of the coppers. The deoxidized high-phosphorus copper was used for this series also, with crosshead speeds (free running) which varied from 0.0059 in. per min to 0.7 in. per min. These tests were conducted at two temperatures—room temperature and 400 F.

Several different methods were used to obtain the desired testing temperatures. For the elevated temperatures, a special 2000-watt electrical tube-type furnace was used. This furnace was capable of providing temperatures up to about 1400 F. For the tests conducted at temperatures between room temperature and −92 F, a testing bath of solvent cooled with dry ice (solid CO_2) was used. The extremely low testing temperatures were obtained with a liquid nitrogen bath.

DISCUSSION AND ANALYSIS OF TEST RESULTS

The static tension tests discussed in this paper were conducted to determine, at various temperatures, the effect of a number of variables on the mechanical properties of three coppers. These variables included the temper, type of material, thickness, direction of rolling, and temperature of testing, each of which will be dealt with in the subsequent sections.

Effect of Temper on the Mechanical Properties:

The word "temper" as used herein refers to the heating and rolling treatment which the material received prior to testing. All of the coppers were tested in two tempers; annealed or cold-rolled (5 to 7 per cent). It should be noted that although the amount of cold rolling was

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[2] 1949 Book of ASTM Standards, Part 1, p. 1233; Part 2, p. 993.

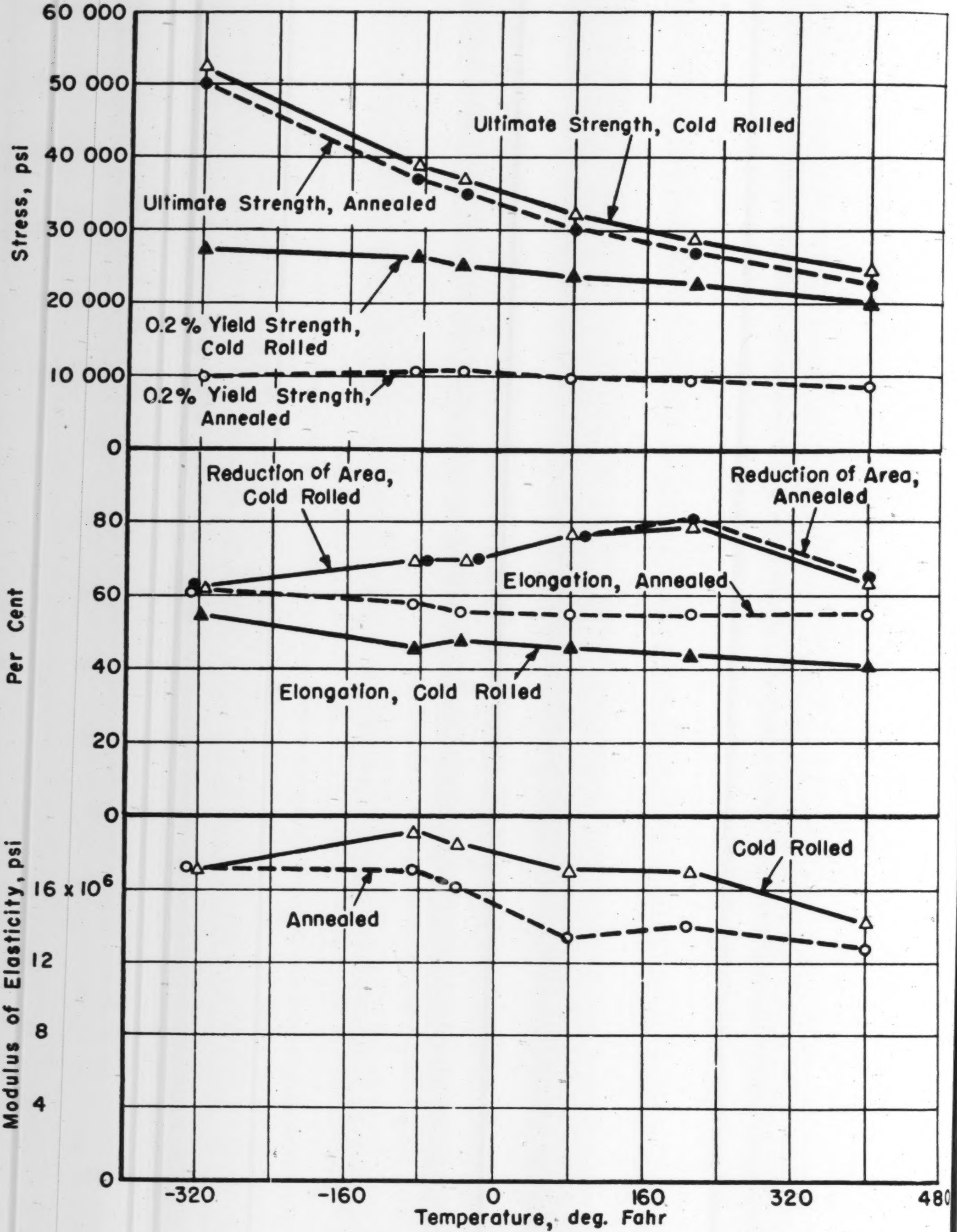

FIG. 1.—Effect of Temper on the Mechanical Properties of Copper at Various Temperatures.

small, the variation between 5 and 7 per cent is relatively large, amounting to a range of 33 per cent. The influence of rolling, as will be pointed out later, varies from one material to another, possibly because of this variation.

The effect of temper on the mechanical properties of the coppers at various temperatures is shown in Fig. 1. Each of the points plotted in this figure represents the average of 24 tests, comprising duplicate tests on three different coppers in two thicknesses and two directions of rolling. It is important also to note that this study of the effect of cold rolling on the mechanical properties is for two conditions only, namely, zero and 5 to 7 per cent reduction in rolling. Although this does not permit broad generalizations concerning the effect of various amounts of cold rolling on the mechanical properties, the trend of the variation of the properties with rolling is readily discernible. The effect of a wider range of cold rolling on the mechanical properties of copper will be dealt with in a subsequent section.

Cold rolling had the greatest effect on the yield strength. The 5 to 7 per cent rolling increased the yield strength of the coppers to a value approximately 2.5 times as great as that of the annealed material. This ratio, however, was affected somewhat by the temperature of the tests. As the testing temperature decreased from 400 F to −321 F, the yield strength of the cold-rolled material increased from 20,000 to 27,000 psi, while that of the annealed material increased from 8500 to 10,500 psi. Furthermore, with decreasing temperatures, the ultimate strength of the coppers of both tempers increased at a considerably greater rate than did the yield strength. Therefore, the relative elastic range of the annealed and cold-rolled coppers, approximated by the ratio of yield strength to ultimate strength, decreased with decreasing temperatures.

The effect of the cold rolling on the ultimate strength was much smaller than on the yield strength. Throughout the range of testing temperatures the cold-rolled material exhibited a consistently higher ultimate strength than did the annealed material. However, the difference was small—averaging between 4 and 7 per cent or approximately 1800 psi over the entire temperature range. The greatest increase in ultimate strength due to rolling was 10 to 15 per cent and occurred for the $\frac{1}{8}$-in. electrolytic copper. This material was undoubtedly subjected to the largest amount of rolling. On the other hand, the ultimate strength of the $\frac{1}{4}$-in. oxygen-free high-conductivity copper was only about 1 to 1.5 per cent higher for the cold-rolled than for the annealed material.

The per cent elongation was generally smaller for the cold-rolled material than for the annealed material. However, for the per cent reduction of area, the temper seemed to have no appreciable effect. This property, as shown in Fig. 1, is further characterized by the fact that, for both tempers, the reduction of area attained its maximum value between 200 to 300 F, and decreased from this point with either increasing or decreasing temperatures.

For the modulus of elasticity, the last property considered in this section, the influence of rolling was small and varied with the testing temperature. However, in general, the modulus of elasticity was higher for the cold-rolled material than for the annealed material.

Effect of Type of Copper on the Mechanical Properties:

For the sake of brevity, the three coppers included in this investigation will

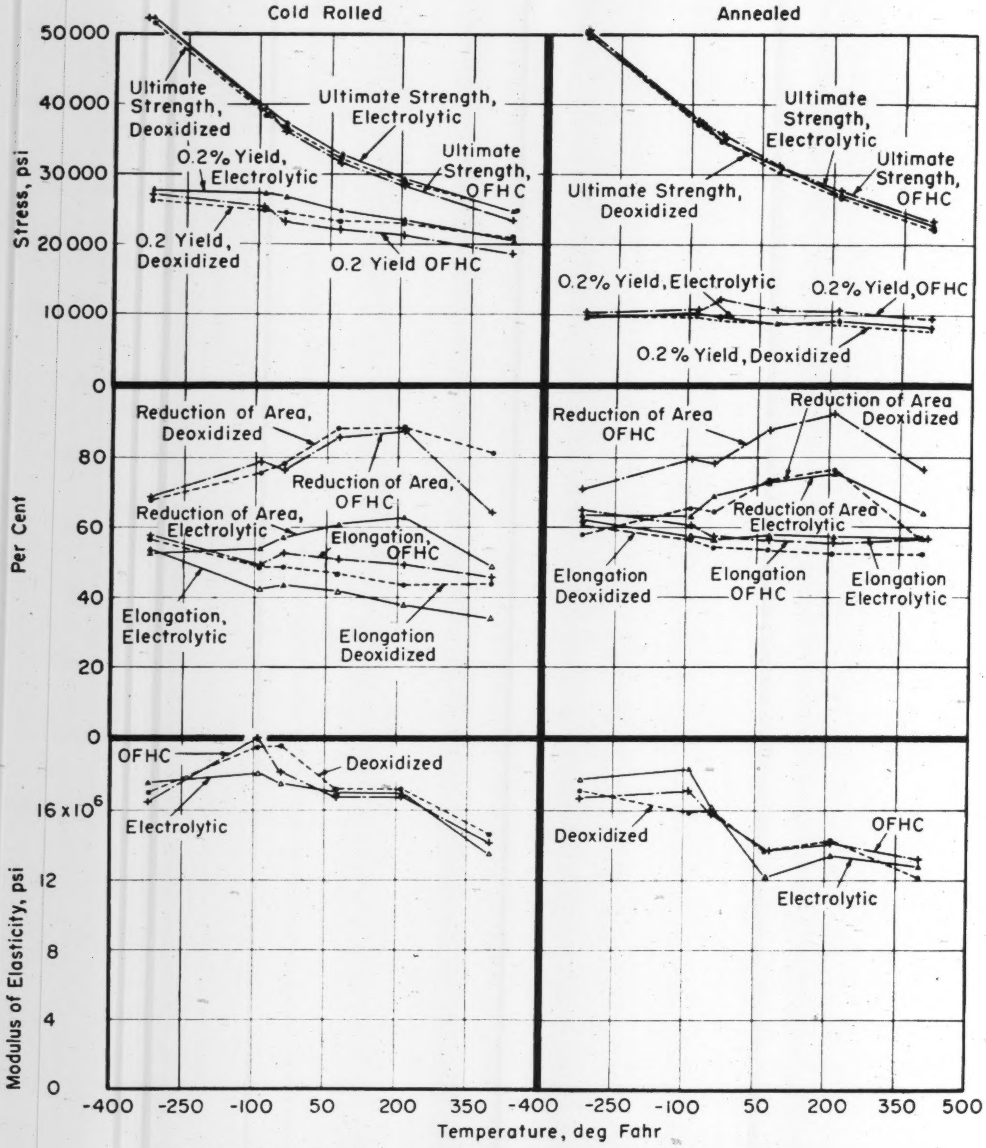

FIG. 2.—Mechanical Properties of Coppers at Various Temperatures.

be designated as electrolytic, deoxidized, and OFHC copper. These coppers vary little in their chemical composition. Each contains more than 99.9 per cent copper, thereby placing them in the class of the "commercially pure coppers."

As might be expected, because of the almost identical chemical compositions, the mechanical properties of these coppers are almost the same. This refers mainly to the strength properties of the coppers; there are noticeable deviations for the ductility properties. Moreover, variables such as the cold rolling, testing temperature, or thickness of material, each seem to affect these three coppers in about the same manner.

The effect of the composition or type of copper on the mechanical properties at the various temperatures is given in Fig. 2. To facilitate the comparisons of the test results, the annealed and cold-rolled coppers have been considered separately in this and several of the following sections. Each of the values plotted in Fig. 2 represents an average of 8 tests—duplicate tests on specimens of two thicknesses and two directions of rolling. By grouping the test results in this manner, the variation of the mechanical properties with temperature for the three types of copper is emphasized.

The differences between the yield strengths of the annealed materials were small and became insignificant at temperatures below −90 F. Nevertheless, the OFHC copper had the highest yield strength throughout the temperature range, and the deoxidized copper the lowest. The differences in the yield strength of the three cold-rolled coppers were more pronounced than for the annealed condition. However, this effect may have been a result of slight differences in the amount of rolling rather than an effect produced by the variation in the compositions of the copper.

The variation in ultimate strength of the annealed and cold-rolled coppers with the type of copper was similar to the variation of yield strength. It was extremely small throughout the entire temperature range studied, not exceeding 1000 psi at any testing temperature, and was, in general, in the same sequence as the yield strength.

For the annealed materials the variation in elongation with the type of copper was quite small. However, for the cold-rolled materials the variation was somewhat larger, diminishing as the temperature was lowered; the difference between the OFHC and electrolytic copper was 11.4 per cent at 400 F and 4.5 per cent at −321 F. The sequence in elongation for the cold-rolled materials was exactly opposite to their sequence in ultimate strength; that is, a smaller elongation was accompanied by a higher strength.

The OFHC copper had by far the highest reduction of area for the annealed materials. In the cold-rolled condition the reduction of area curves for the deoxidized and the OFHC coppers follow each other closely throughout most of the temperature range, but the curve for the electrolytic copper is generally 15 to 20 per cent below that of the other two coppers.

The curves of Fig. 2 show that the modulus of elasticity decreased for both the annealed and cold-rolled materials as the temperature increased above −90 F. In addition, it is noted there was no consistent effect of the type of material on the value of the modulus of elasticity for the three coppers.

Effect of Thickness on the Mechanical Properties:

Since this investigation included tests on specimens of two thicknesess, a study has been made to determine the relationship between the mechanical properties of the coppers in these thicknesses

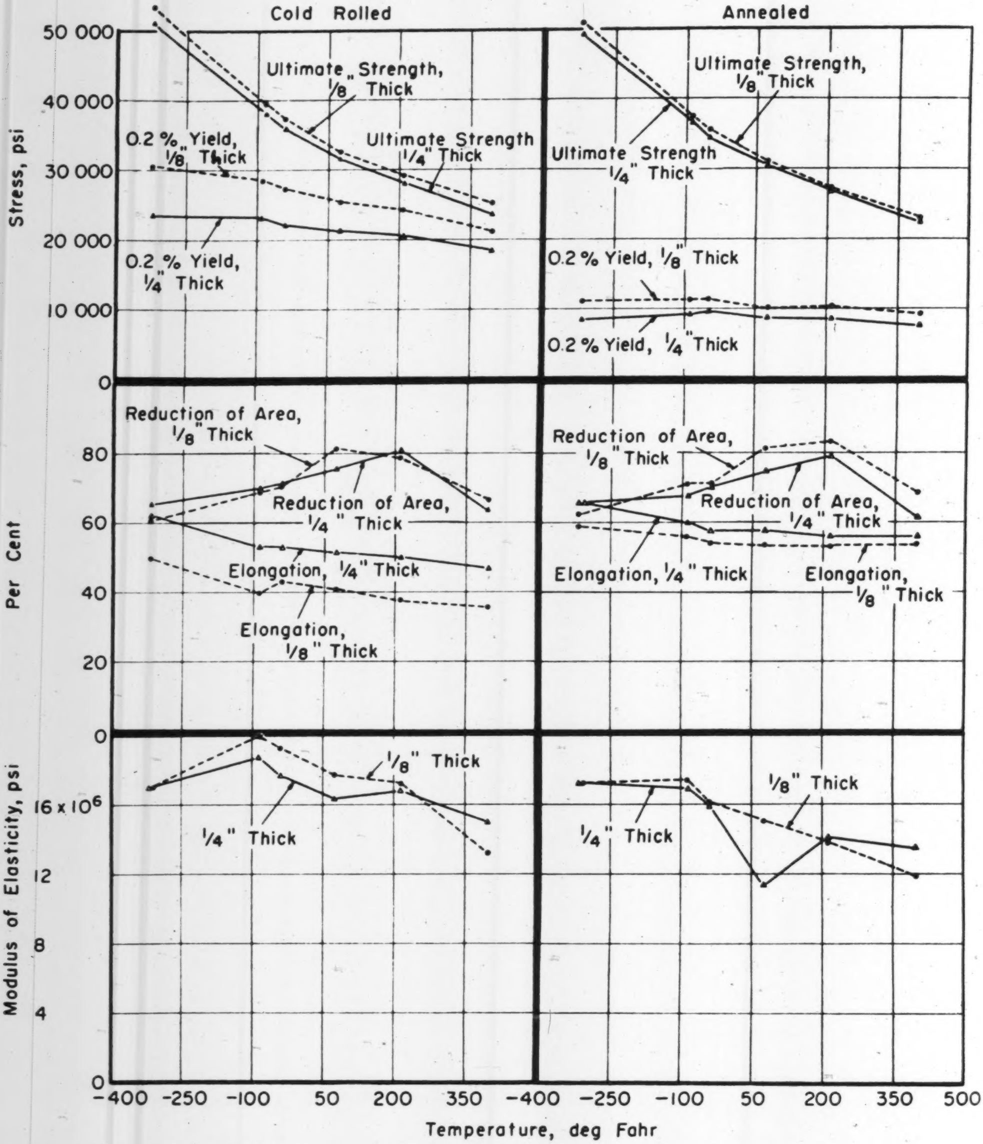

FIG. 3.—Effect of Thickness of Material on the Mechanical Properties of Copper at Various Temperatures.

(see Fig. 3). The yield strength of the annealed coppers was found to be 12 to 20 per cent higher for the $\frac{1}{8}$-in. specimens than for the $\frac{1}{4}$-in. specimens. This applies also to the variation in yield strength of the cold-rolled material; the $\frac{1}{8}$-in. material had a yield strength 14 to 30 per cent greater than that of the $\frac{1}{4}$-in. material.

The variation in ultimate strength with a change in thickness was smaller but similar to that found for the yield strength. In both the annealed and cold-rolled conditions the $\frac{1}{8}$-in. material had a greater strength than the $\frac{1}{4}$-in. material throughout the entire temperature range.

The elongation of the $\frac{1}{4}$-in. annealed material was somewhat greater than that of the $\frac{1}{8}$-in. annealed material. The same, in general, applies in the case of the cold-rolled specimens also, except that the effect of thickness was more pronounced for the cold-rolled material than for the annealed coppers. Throughout the temperature range the curves for the elongation of the $\frac{1}{4}$-in. and $\frac{1}{8}$-in. materials are almost parallel, but the $\frac{1}{4}$-in. material elongated 10 to 14 per cent more than the $\frac{1}{8}$-in. material.

The effect of thickness on the reduction of area of the annealed and cold-rolled coppers is not as well defined as are some of the other effects. However, in general it appears that at the higher temperatures the $\frac{1}{8}$-in. material had greater values of reduction of area than did the $\frac{1}{4}$-in. material, and at the lower temperatures the reduction of area for the $\frac{1}{4}$-in. material was slightly greater.

No consistent trend or definite effect of thickness on the modulus of elasticity could be discerned for the coppers in the annealed state;[3] but in the case of the cold-rolled coppers, the $\frac{1}{8}$-in. material, in general, had higher values for the initial slope of the stress-strain curves than did the $\frac{1}{4}$-in. material.

Effect of the Orientation of Specimen with Respect to Direction of Rolling on the Mechanical Properties:

The coupon specimens were tested in two directions of rolling; their longitudinal axis was either parallel or perpendicular to the direction of rolling. In this and the following sections, specimens referred to as longitudinal are those whose longitudinal axis was parallel to the direction of rolling, whereas transverse refers to those specimens whose longitudinal axis was perpendicular to the direction of rolling. Specimens of each type of material, thickness and temper, were tested in both of these directions.

To emphasize the effect of the direction of rolling on the mechanical properties of the coppers, the test results for specimens of the three coppers and two thicknesses have been averaged. Figure 4 presents the results of these comparisons.

The yield strength of the annealed coppers was found to be nearly the same for the transverse specimens and the longitudinal specimens. However, for the cold-rolled materials, the longitudinal specimens had a yield strength approximately 2000 to 3000 psi higher than that of the transverse specimens. This would seem to indicate that the cold rolling is responsible for the variation in yield strengths between the transverse and longitudinal specimens.

The ultimate strength of the annealed coppers, as in the case of the yield strength, was nearly the same for the transverse and longitudinal specimens. On the other hand, the effect of the direction of rolling on the ultimate strength of the cold-rolled copper was noticeable; throughout the temperature range, the longitudinal specimens had a higher strength than did the transverse

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[3] The low value for the $\frac{1}{4}$-in. annealed material tested at room temperature is believed to be due mainly to difficulties encountered with the test equipment in the early stages of the program.

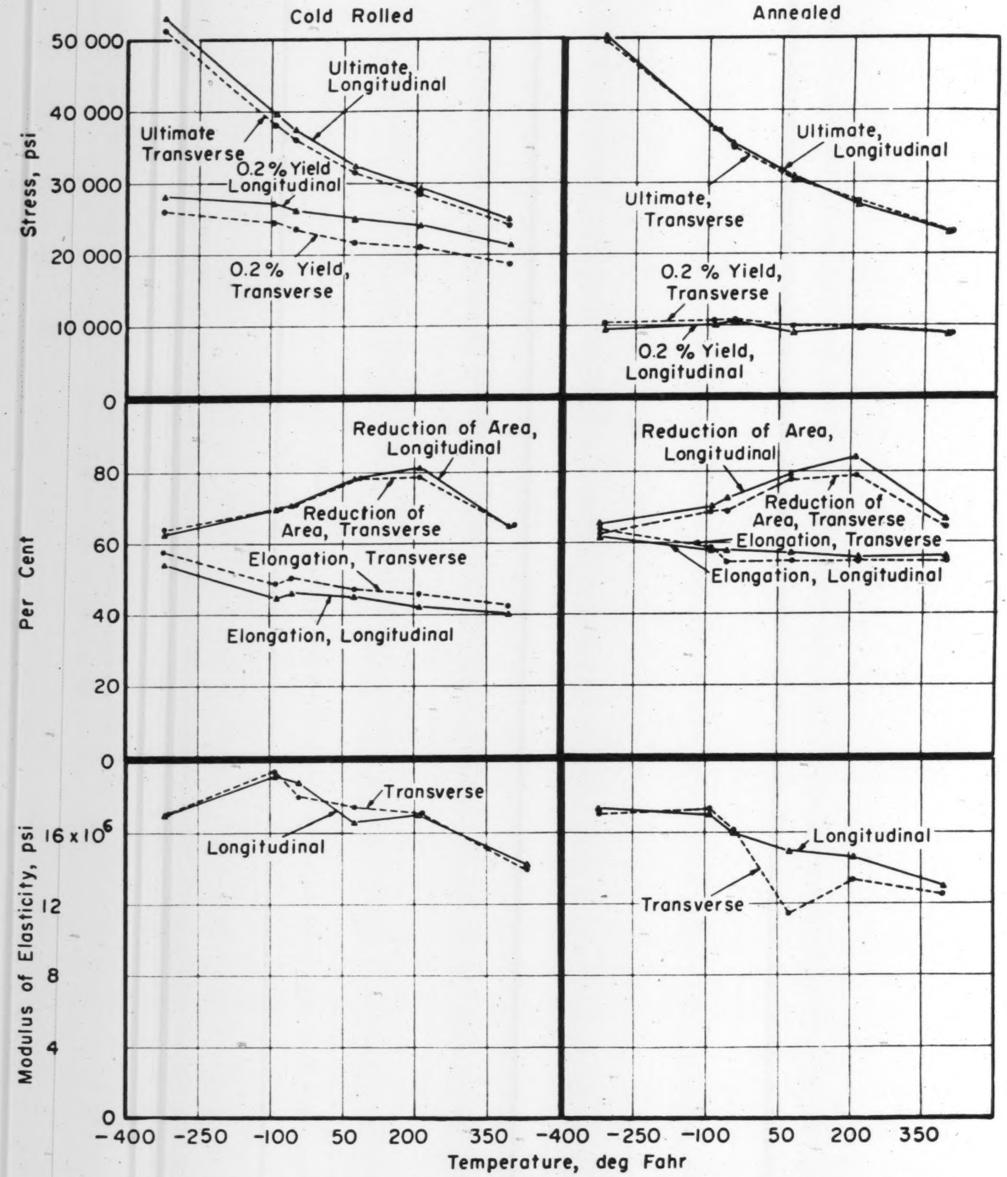

FIG. 4.—Effect of Direction of Rolling on the Mechanical Properties of Copper at Various Temperatures.

specimens. This difference in strength, however, decreased from 1650 psi at −321 F to 700 psi at 400 F.

The direction of rolling seemed to have little or no effect on the elongation of the annealed material. Values of elongation were nearly the same for both the longitudinal and transverse specimens. In the case of the cold-rolled material, the specimens in the longitudinal direction always had less elongation than the transverse specimens. Again it appears that the cold rolling is the factor which causes the variations between the longitudinal and transverse specimens.

The reduction of area of the longitudinal specimens was consistently higher than that of the transverse specimens for the annealed materials. For the cold-rolled material, the variation in reduction of area resulting from the direction of rolling was negligible. However, as will be discussed in the next section, this property is greatly affected by the type of fracture and for that reason it is difficult to draw definite conclusions from the reduction of area curves without taking into account the type of fracture.

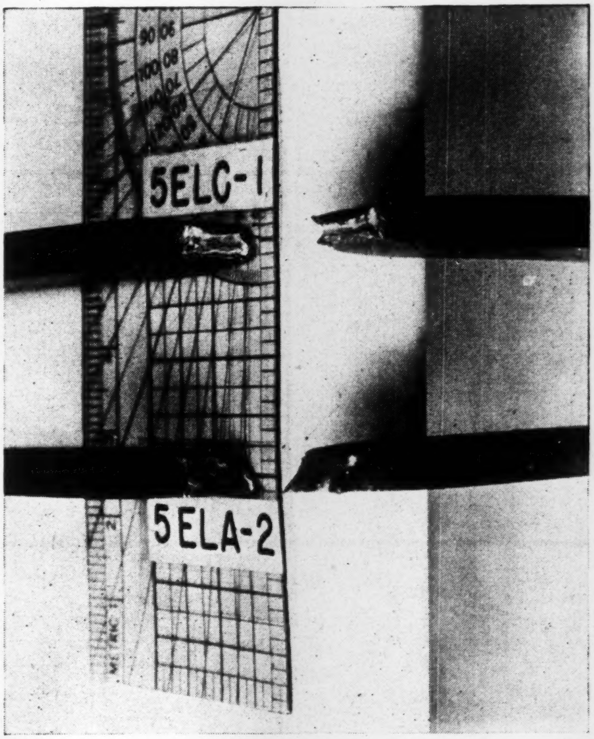

FIG. 5.—Single Shear and Cup-Type Fractures of Copper Specimens.

Because of the somewhat inconsistent test values obtained at room temperatures,[3] the influence of the direction of rolling on the modulus of elasticity of the coppers is not too well defined. As shown in Fig. 4, there was, in general, no consistent variation in the modulus of elasticity with the direction of rolling for either the annealed or cold-rolled material.

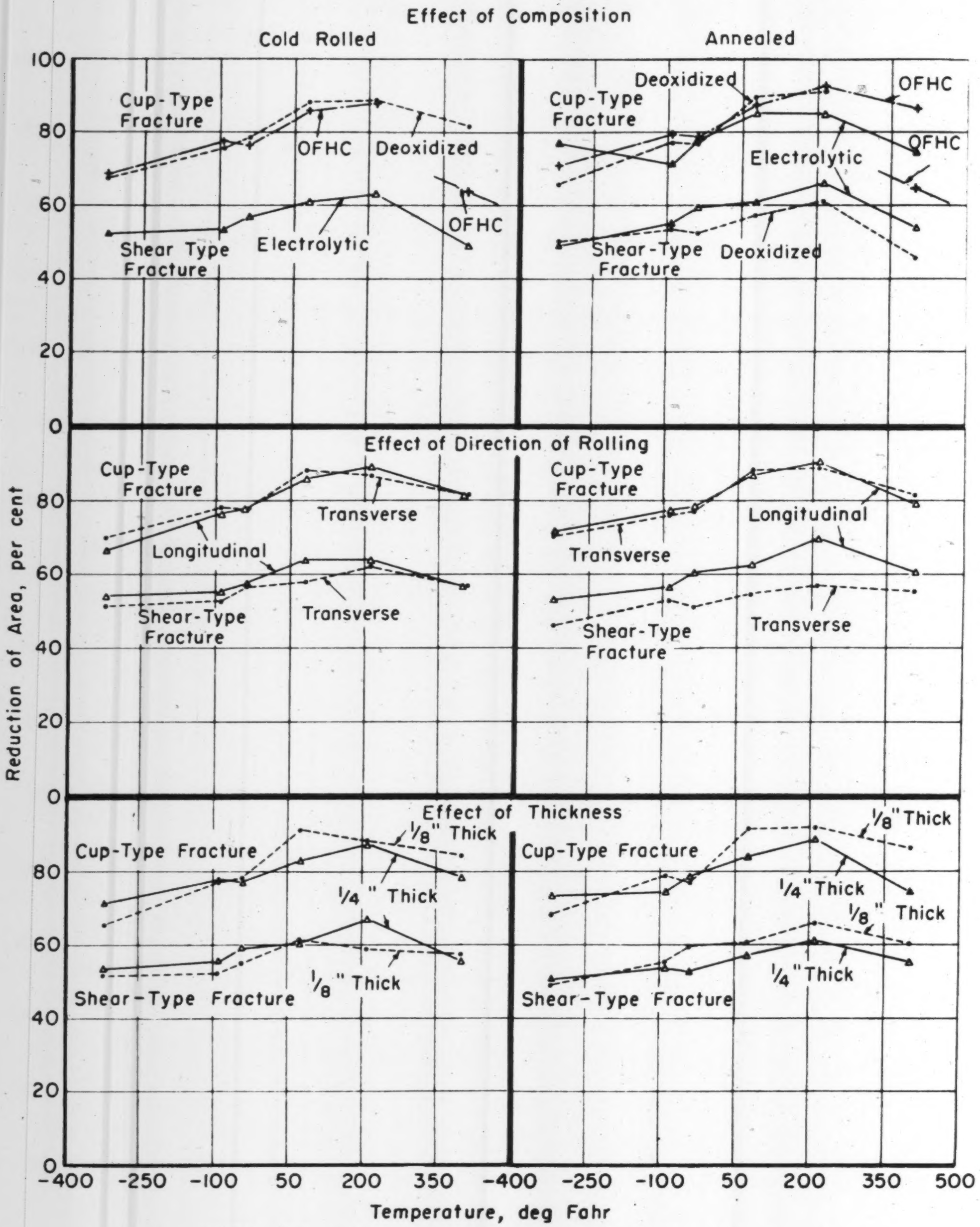

FIG. 6.—Effect of Type of Fracture on the Reduction of Area.

Effect of Type and Location of Fracture on the Mechanical Properties:

A detailed examination of the fractures of the specimens disclosed that the type of fracture had a pronounced effect on the elongation and reduction of area of the coppers. Two distinct types of fractures were obtained: single plane shear-type or a double plane cup-type. Both of these were strictly ductile fractures and occurred after a considerable amount of elongation and with no tendency for brittle fracture within the temperature range of −321 to 400 F. The two types of fractures could be easily distinguished by the appearance of the fracture surface, as shown in Fig. 5. The shear-type fracture occurred on a single 45-deg shear plane and was characterized by a smooth fracture surface with straight edges. The cup-type fracture was essentially a double-shear failure accompanied by a large amount of localized necking at the fracture section.

The results of the study on the effect of the type of fracture on the reduction of area are presented in Figs. 6 and 7. It is evident immediately in Fig. 6 that the type of fracture had a much greater effect on the ductility properties than did the type of copper, direction of rolling or thickness. The three coppers tested responded in different ways with respect to the type of fracture. The electrolytic copper was more apt to fail in a shear type manner. The only specimens of this material that failed with a cup-type fracture were the annealed ¼-in. specimens. The deoxidized copper was exactly opposite and displayed a cup-type fracture except for the annealed ¼-in. specimens, while all of the specimens of OFHC copper had a cup-type fracture except for those tested at 400 F. In general the temperature did not seem to have

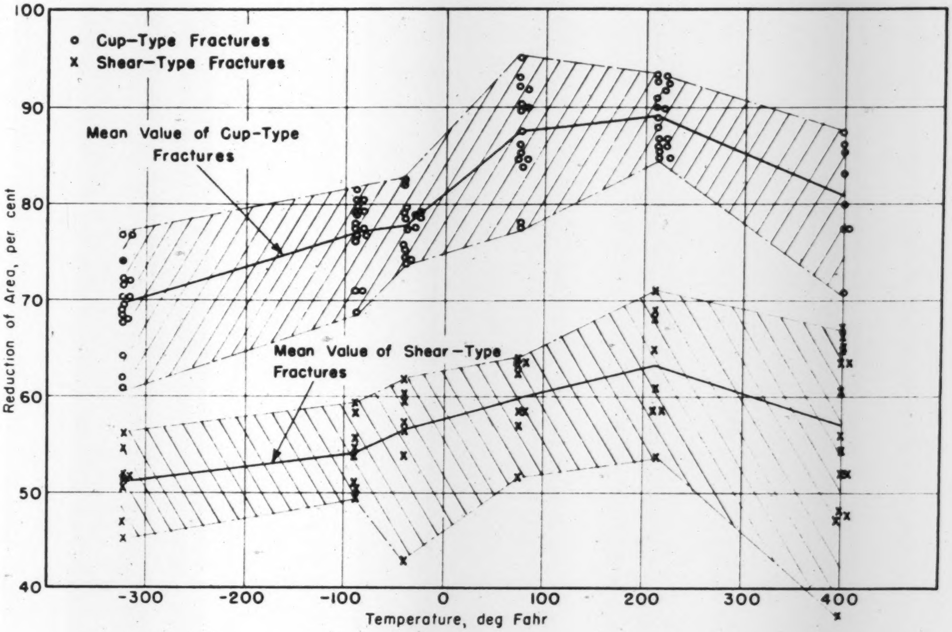

Fig. 7.—Average Reduction of Area for Cup and Shear-Type Fractures.

any effect upon the type of fracture; however, at 400 F a peculiar phenomenon occurred: some of the materials which generally failed in a cup-type manner failed with a shear-type fracture. This applies mainly to the OFHC copper and to a lesser degree to the deoxidized copper. It is significant also that regardless of the type of fracture, each individual material had a maximum value for reduction of area at about 200 to 300 F.

The various other factors considered in studying the effect of the type of fracture on the reduction of area show, in general, the same characteristics as the effect of composition discussed above. The effects of the different variables are either small or non-existent when compared with the effect of the type of fracture. This is particularly true for the effect of the direction of rolling or thickness of specimen on the reduction of area values for the various coppers.

Regardless of thickness, orientation, temper or composition of copper, the values of reduction of area for the shear-type and cup-type fractures fell into two separate bands throughout the temperature range of the tests. These bands are shown in Fig. 7 along with the mean value for each type of fracture. The two average curves are roughly parallel with the mean value curve for the cup-type fractures about 40 per cent higher than that for the shear-type fractures.

Aside from its effect on the reduction of area, it was considered plausible that the type of fracture might affect also the elongation, since the cup-type fracture generally exhibited more local strain at the failure than did the shear-type fracture. This picture, however, is complicated by the effect of the location of the fracture. As the position of fracture approaches the end of the gage length, more of the highly strained material falls outside the gage length and the apparent elongation values are reduced. An examination with regard to the location of fracture indicated that a slight but gradual decrease of elongation occurred for the cup-type fractures as the location of fracture approached the ends of the gage length. A less defined and more erratic decrease was evident also for the shear-type failures but this was partially overshadowed by local irregularities. Nevertheless, the shear-type fracture gave a lower value of elongation for a given location of fracture than did the cup-type fracture.

TABLE I.—EFFECT OF CROSSHEAD SPEED OF TESTING MACHINE ON THE MECHANICAL PROPERTIES OF COPPER AT ROOM TEMPERATURE AND 400 F.

Temperature of Test, deg. Fahr.	Speed of Crosshead, in. per min*	Mechanical Properties			
		Yield Strength, 0.2 per cent offset, psi	Ultimate Strength, psi	Reduction of Area, per cent	Elongation in 2 in., per cent
75	0.0059	25 110	32 500	78.7	44.5
	0.0345	25 400	32 810	77.8	48.6
	0.119		33 200	78.0	48.8
	0.7		33 900	79.1	50.5
400	0.0059	21 050	23 700	75.6	46.0
	0.0345	22 400	24 560	77.5	47.6
	0.119		25 140	79.2	45.5
	0.7		25 430	78.4	48.5
VALUES OBTAINED FOR THE MAIN TEST SERIES. MATERIAL SAME AS ABOVE.					
75......	0.0059–0.119	25 250	33 160	84.6	52.0
400......	0.0059–0.119	22 090	25 170	80.0	46.5

* Free-running speed.

Effect of Speed of Loading on the Mechanical Properties:

The tension tests reported in this paper were all started at a slow testing speed (0.0059 in. per min free-running crosshead speed) to permit load and elongation readings to be taken without stopping the testing machine. In order to expedite the tests, the cross head speed was increased to 0.119 in. per min after an elongation of about 1½ per cent had been reached. Consequently, the modulus of elasticity and yield strength were obtained at the slower testing speed and the ultimate strength, elongation in 2 in., and reduction of area were obtained after the speed of testing had been increased. Because of this variation in rate of loading, a short series of tests was included in the test program to determine the effect of the speed of loading on the mechanical properties of the coppers.

This additional series of tests was conducted on longitudinal specimens of $\frac{1}{4}$-in. cold-rolled deoxidized high-phosphorus copper at four crosshead speeds (free-running) ranging from 0.0059 in. per min to 0.7 in. per min. These four speeds of loading (see Table I) represent the variations in the rate of crosshead movement possible with the 30,000-lb testing machine used for the tests in this program.

A total of 22 specimens were tested, 11 at room temperature and 11 at 400 F, each with the testing machine in continuous operation at the desired speed. With this method of testing, it was difficult to obtain accurate strain readings in the elastic range of the material at the 0.119 in. per min and 0.7 in. per min speeds of the testing machine.

From the test data it is evident that the ultimate strength increased slightly as the strain rate was increased. However, the increase in ultimate strength for the range of strain rates used in this investigation amounted to only 1400 psi at room temperature and 1730 psi at 400 F. This is in agreement with the results of the tests reported by Manjoine and Nadai (**19**, **22**)[4] for comparable testing speeds. Therefore, it was concluded that the reported mechanical properties were little affected by the shift in loading speed made after an elongation of $1\frac{1}{2}$ per cent and that comparisons of mechanical properties reported by different investigators will not be greatly affected as long as the speed of testing does not exceed that generally used for static tests.

For comparison, the values obtained in the main test series for the $\frac{1}{4}$-in. deoxidized copper are also given in Table I. Because of the change in speed during these tests, the values for the yield strengths correspond to the slowest speed of testing (0.0059 in. per min) and the values of the ultimate strength, reduction of area, and elongation in 2 in. correspond to a crosshead speed of 0.119 in. per min. It can be seen that these values are in close agreement with the other data obtained at the corresponding speeds.

Mechanical Properties of Deoxidized High-Phosphorus Copper at Temperatures from −321 to 1200 F:

Because of the large interval between the test temperatures and the relatively low maximum temperature one of the coppers was tested over a temperature range of 400 to 1200 F and at a number of additional intermediate temperatures between −321 and 400 F. These tests, along with the tests of the regular testing program, have been combined for the studies presented in this section.

The $\frac{1}{4}$-in. deoxidized high-phosphorus cold-rolled copper loaded parallel with the direction of rolling was selected for this study. Three identical coupon specimens were tested at each of the intermediate temperatures and two were tested at each of the higher temperatures (see Fig. 8).

Values of the yield strength (0.2 per cent offset method) and ultimate strength are shown in the upper part of Fig. 8. The decrease in the yield strength and ultimate strength, with increasing temperatures, can be readily observed. For the yield strength this was most pronounced at the higher temperatures (300 F to 850 F). At temperatures in the

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[4] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1018.

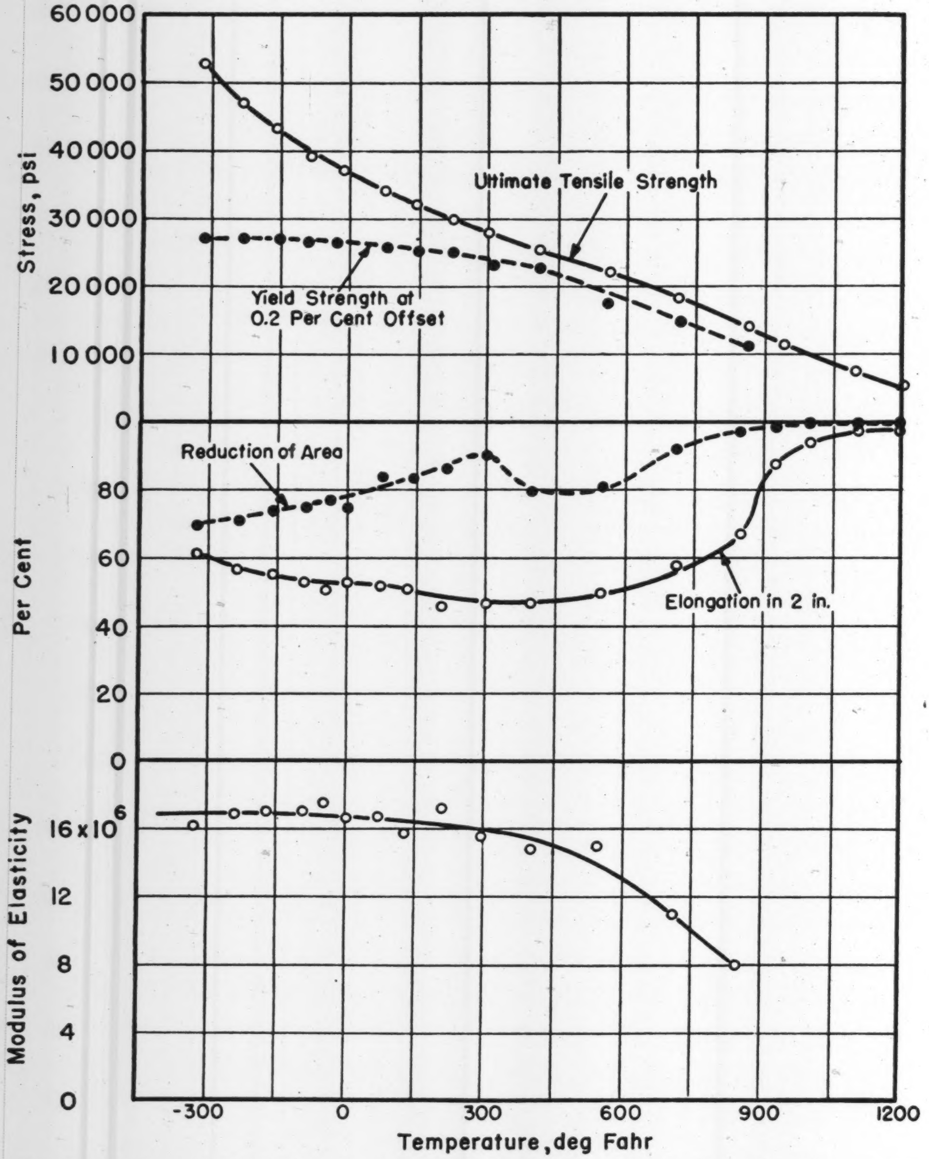

FIG. 8.—Mechanical Properties of a Cold-Rolled, $\frac{1}{4}$-in. Deoxidized High-Phosphorus Copper at Various Temperatures.

range from −40 to 300 F, the drop in yield strength was small and below −40 F. the yield strength was practically constant.

The variation of the ultimate strength with respect to temperature was the most uniform of the mechanical properties considered. Except for the lower end of the temperature range, the data can be represented approximately by a straight line. However, below a temperature of about 0 F the ultimate strength increased at a greater rate than at the higher temperatures. The ultimate strength was 52,300 psi at −321 F and 4800 psi at 1200 F, the highest temperature used ln these tests.

Figure 8 includes also curves which show the variation in reduction of area and elongation with temperature. The reduction of area increased as the temperature was increased from −321 F and reached a peak of 90 per cent at 300 F. This was followed by a drop to 80 per cent reduction of area at 400 F and then an increase to almost 100 per cent at 900 F. At this temperature and above, the fracture surface was reduced to a sharp edge rather than a fracture area. The thickness of a fracture obtained at 1000 F was checked under a microscope and found to be about 0.001 in. at the thickest point.

The type of fracture did not affect the result of the temperature series, since the type of copper tested in this series exhibited a cup-type fracture at all of the testing temperatures. This is compatible with the results obtained in the regular test series.

The per cent elongation of the deoxidized copper decreased from 61.5 per cent at −321 F to approximately 45.0 per cent at 300 F and then increased to 60 per cent at about 750 F. An abrupt increase in elongation occurred between 750 and 1100 F and from 1100 F to 1200 F it seemed to remain constant at about 98 per cent.

The influence of the high temperatures on the ductility was not only indicated by the high values of the reduction of area and elongation, but also by the marked decrease in cross-sectional area along the entire length of the specimen; sharp necking which is so pronounced at lower temperatures was hardly perceptible as the material elongated excessively. This phenomenon could also be observed, but to a lesser degree, at 850 F and 925 F.

The modulus of elasticity was approximately constant over the temperature range from −321 to 300 F at 17 × 10^6 psi. Beyond 300 F, however, the modulus of elasticity decreased sharply as the temperature increased and at 850 F was equal to about 8 × 10^6 psi. These results are similar but somewhat lower than those reported by Köster (30) for an extremely pure copper.

The extremely low temperatures did not impair the mechanical properties of the copper; in fact, with the exception of the reduction of area, all of the mechanical properties either stayed constant or showed a tendency to increase as the temperature was lowered. A definite increase was obtained in the yield strength, ultimate strength, and the elongation, indicating that the energy absorbing capacity also increased with a decrease in temperature. Conversely, the influence of elevated temperatures is characterized by a drop in the ultimate strength and yield strength as the temperature approaches the melting point of copper.

COMPARISON OF TEST RESULTS WITH PREVIOUS INVESTIGATIONS

In the 1924 ASTM-ASME Symposium on "The Effect of Temperature upon the Properties of Metals," Upthegrove and White (3) presented a complete discussion of the data available at that time, on the properties of copper at various temperatures. This was followed by several similar Symposiums in 1931 (6), 1939 (23), and 1946 (28). In 1939, a review of the literature from 1931 to 1939 was given by Smith (14) in his study of the mechanical properties of copper and its alloys at low temperatures.

Comparisons of the results of the tests reported herein with data compiled by previous investigators are plotted in Figs. 9 to 13 inclusive and are discussed in detail in the following sections.

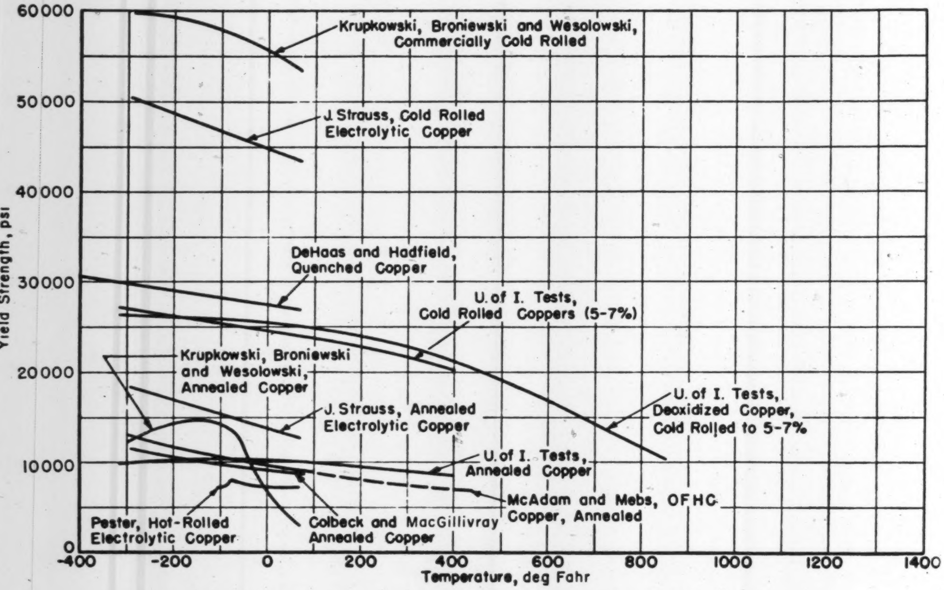

FIG. 9.—Effect of Temperature on the Yield Strength of Various Coppers.

Effect of Temperature on the Yield Strength of Various Coppers:

Figure 9 shows the effect of temperature on the yield strength of various coppers as obtained from tests by a number of investigators. It is apparent that the yield strength increases with a decrease in temperature for a given material and with an increase in cold rolling at a given temperature. The yield strength of the annealed coppers as obtained by Colbeck and MacGillivray (11), McAdam and Mebs (25), and by the authors are grouped closely together. Krupkowski (8) and Broniewski and Wesolowski (12) report a maximum yield strength at about −110 F and an extremely low yield strength at room temperature for an annealed copper. The yield strength reported by Strauss (5) for annealed electrolytic copper appears to be high compared with the values for the other annealed coppers shown. DeHaas and Hadfield (13) reported values of yield strength which are almost 200 per cent higher than those obtained in the other investigations for annealed materials; however, the material they tested was a copper quenched from 1382 F. It is questionable also whether the drop of beam method, as applied by DeHaas and Hadfield to obtain the yield strength, is acceptable for this type of material. Another factor or variable which may have an effect on the results shown in this figure is the shape and size of the test specimens. However, this is not taken into account in the comparisons presented herein.

Strauss (5), and Krupkowski and his coworkers (8), present results which show the same general trend for cold-rolled coppers as mentioned above for the annealed coppers. It is difficult, however, to determine how great an effect the rolling has on the yield strengths for the materials shown since the amount of rolling is unknown except for the tests reported in this paper. The materials for the University of Illinois tests were rolled to a 5 to 7 per cent reduction in area; nevertheless, this small amount of rolling provided a yield strength approximately 150 per cent greater than that obtained for the corresponding annealed materials.

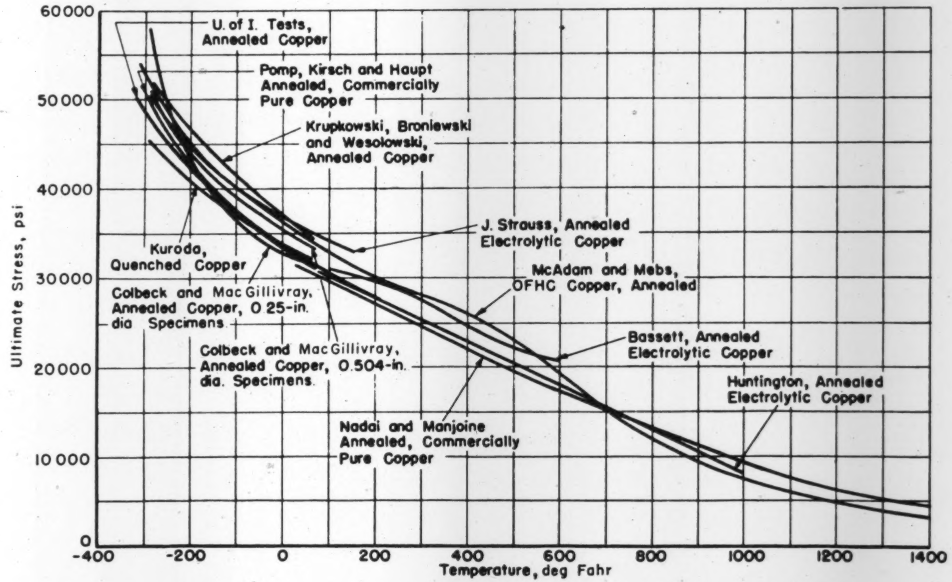

FIG. 10.—Effect of Temperature on the Ultimate Strength of Various Annealed Coppers.

Effect of Temperature on the Ultimate Strength of Various Coppers:

A number of investigators have obtained the ultimate strength of annealed coppers at various temperatures. The results of their studies are presented in Fig. 10 and show excellent agreement. The width of the band of the reported values for various annealed coppers does not exceed, in general, 5000 psi. At temperatures from 0 to 900 F, the variation of ultimate strength is approximately linear. Beyond this point it gradually approaches the zero strength axis as the temperature nears the melting point of the metal (1980 F). Creep starts to play a critical rôle at approximately 400 F, so that values reported for temperatures above 400 F will depend, to some extent, upon the duration of the test and the magnitude of the temperature. Below 0 F a progressive increase occurs in the ultimate strength as the temperature decreases.

The effect of temperature combined with cold rolling is shown in Fig. 11. These curves are similar and approximately parallel at temperatures below 200 F; however, the materials with a greater amount of rolling have a much higher ultimate strength. Although the influence of rolling is not too clear in the figure, it appears that the greatest increase in ultimate strength occurs for a rolling between 6 and 25 per cent. As the temperature exceeds 300 F, the effect of cold-rolling is gradually removed by annealing, and the ultimate strength approaches that of the annealed coppers. The sharpest drop in ultimate strength for the cold-rolled coppers occurs between 400 and 600 F. Above the recrystallization temperature, about 800 F, no difference in strength can be discerned between the annealed and cold-rolled materials.

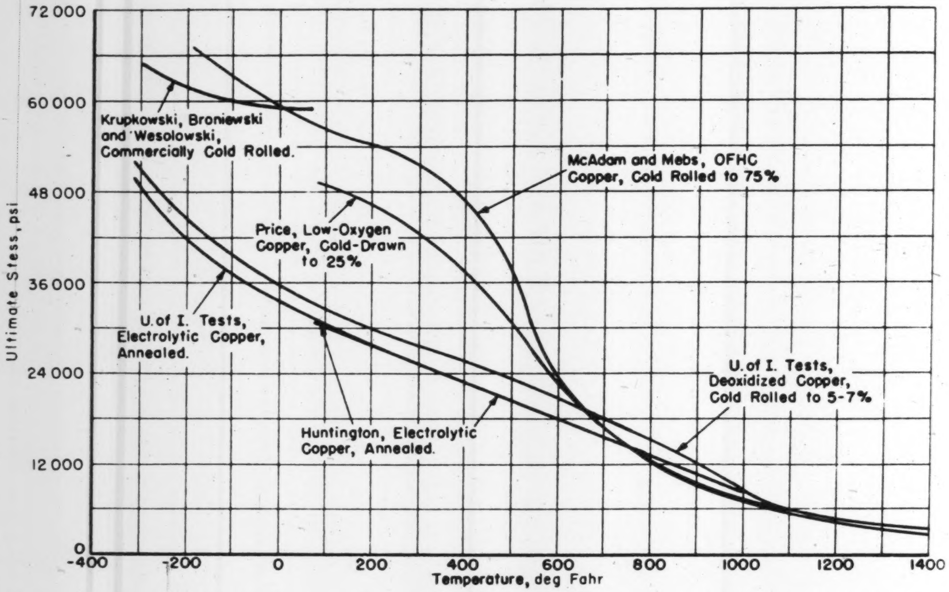

FIG. 11.—Effect of Cold-Rolling on the Ultimate Strength of Coppers at Various Temperatures.

Effect of Temperature on the Elongation of Various Coppers:

The variation of per cent elongation of coppers with temperature, as obtained by various investigators, is shown in Fig. 12. These curves cover mainly the range of temperatures below room temperature. However, a few of the investigators have included also tests at the higher temperatures. Below atmospheric temperature the elongation either stays constant or increases slightly as the temperature is reduced to that of liquid nitrogen (−321 F). There is no evidence to indicate that copper loses its ductility at the low temperatures. It is of interest also, to note that the curves are generally arranged in order by the amount of cold rolling to which the material was subjected. For the tests shown, the elongations range from about 15 to 60 per cent at atmospheric temperature.

The shape of the elongation curves

above 300 F may depend upon such factors as the composition or temper of the material and the atmosphere in which the tests are conducted. In an atmosphere of carbon dioxide, electrolytic copper tested by Bengough and Hanson (2) had elongation properties quite different from those obtained with tests in air.

Price's (6) data for a cold-drawn low-oxygen copper and the data for deoxidized copper obtained in this program show a definite similarity in that both materials exhibit a sudden increase in elongation starting at 400 to 500 F. The slight difference in shape and the rather large difference in the general location of the curves is, no doubt, the result of the difference in the cold rolling of the two materials. It appears that, for high purity coppers such as those tested by Price and by the authors, a large ductility is obtained at temperatures in the range of 800 to 1200 F. The minimum ductility for these two materials occurred at a temperature of about 400 F.

Effect of Temperature on the Reduction of Area of Various Coppers:

In Fig. 13 the variation of the reduction of area with temperature appears to be quite erratic at subatmospheric temperatures. Some investigators have reported increases and others have reported decreases with a decrease in temperature. However, the annealed materials, in general, exhibit a greater reduction of area than the cold-rolled materials within the entire temperature range considered. Another factor which has a large effect upon these results is the type of fracture. The average reduction of area curves for the University of Illinois tests could be raised or lowered by approximately 10 per cent if only the cup or shear type of fracture is consid-

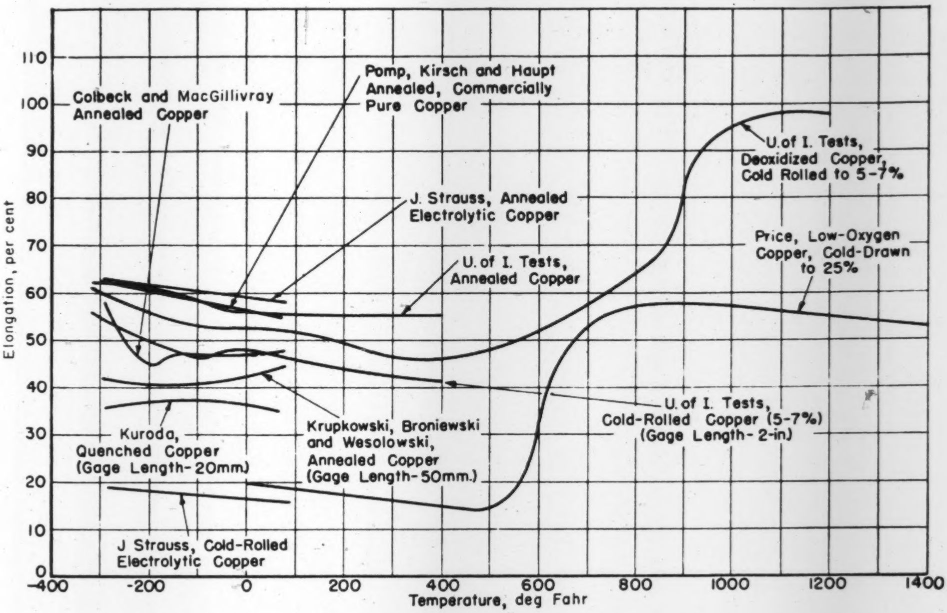

Fig. 12.—Effect of Temperature on the Elongation of Various Coppers.

ered. For this reason comparisons of this property are rather uncertain and must be made with care.

The curves obtained by Price (6) and the authors demonstrate that the low-oxygen and deoxidized coppers have a local peak value of reduction of area at about 300 F followed by a small drop which ends in a local minimum at approximately 450 F. As the temperature rises above 450 F, the reduction of area increases substantially, approaching a 100 per cent value at the highest temperatures. The difference in amounts of cold rolling for Price's material and the copper investigated by the authors accounts for the fact that the reduction curves for these materials are separated by as much as 30 per cent. At the higher temperatures the effects of cold rolling are gradually removed, and the difference between the reduction of area of Price's copper drawn to 25 per cent and the authors 5 to 7 per cent cold-rolled copper gradually disappears; however, at the lower temperatures the shapes of the two curves are almost identical.

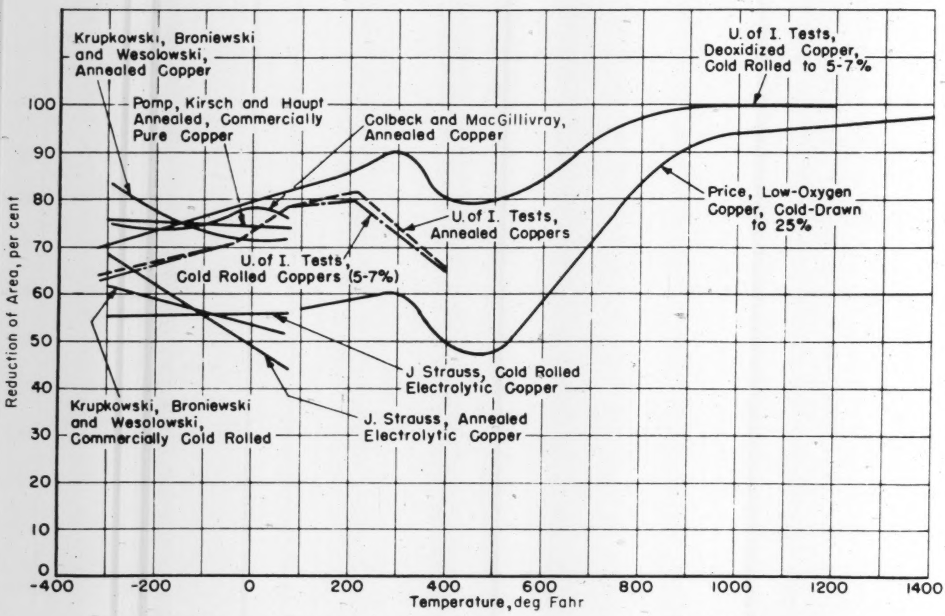

FIG. 13.—Effect of Temperature on the Reduction of Area of Various Coppers.

SUMMARY OF RESULTS AND CONCLUSIONS

The results of the tests discussed in this paper have been presented with two objectives in mind: (1) to show the effect of testing temperature on the mechanical properties of several coppers, and (2) to show the effects of such variables as temper, thickness, composition, and direction of rolling on the mechanical properties of the coppers.

The property most affected by the variation in temperature was the ultimate strength. For a change in testing temperature from 1200 F to −321 F, the ultimate strength increased from about 5000 psi to approximately 50,000 psi. However, the rate of change was greatest at temperatures below atmospheric. The yield strength increased also as the temperature was lowered but, in this case, the rate of change decreased as the temperature was reduced below atmospheric. As a result, the relative elastic range for the coppers decreased at the lower temperatures.

The reduction of area for the various coppers was found to increase gradually as the temperature was increased, except in the region of 300 to 700 F. In this range there was a drop of 10 to 15 per cent in the reduction of area. The other ductility property, elongation, is not greatly affected by an increase in testing temperature until a temperature of about 500 F is reached. At this point a considerable increase in elongation started to take place.

Cold rolling, even in small amounts, changed some of the mechanical properties of the coppers materially. This change was most noticeable for the yield strength: a 5 to 7 per cent reduction in rolling resulted in an increase of approximately 150 per cent. However, an increase of only 4 to 7 per cent was noted in the ultimate strength as a result of the rolling. While the values of these properties were increasing, the elongation decreased and the reduction of area remained almost constant.

The differences in the mechanical properties of the three types of copper were, in general, negligible. However, this should be expected since their chemical compositions are almost identical.

A reduction in the thickness of the coppers, from $\frac{1}{4}$ in. to $\frac{1}{8}$ in., resulted in a pronounced increase in the yield strength (12 to 30 per cent), a somewhat smaller increase in the ultimate strength and a decrease in the elongation. As in the case of cold rolling, an increase in the strength properties was accompanied by a decrease in elongation. On the other hand, this same variation in thickness seemed to have little or no effect on the reduction of area.

The direction of rolling had little effect on the properties of the annealed coppers. However, even a small amount of rolling, such as that used in this study, resulted in a greater strength and a lower elongation for the specimens parallel to the direction of rolling than for the specimens transverse to the direction of rolling. It is evident, therefore, that the properties of a cold-rolled copper will not be uniform in all directions.

Acknowledgments:

The work reported in this paper was part of the research program of the Civil Engineering Department of the University of Illinois and was sponsored by the Copper and Brass Research Assn. The project was started under the general direction of Wilbur M. Wilson, Research Professor of Structural Engineering, Emeritus. Since Professor Wilson's retirement the program has been under the general direction of N. M. Newmark, Research Professor of Structural Engineering.

Acknowledgment is gratefully made to Arthur I. Heim, Engineer of the Copper and Brass Research Assn. for his valuable help in coordinating this program; to Robert J. Mosborg, Research Associate in Civil Engineering for his assistance in developing the special low temperature equipment; and to Robert M. Smith and Wyck McKenzie for their assistance in conducting the tests.

REFERENCES

These references are intended to supplement the references published with the 1924, 1931, 1939, and 1946 Symposiums of the ASTM-ASME Joint Committee on the Effect of Temperature on the Properties of Metals. Several of the references, however, have been repeated herein in order that the reader can refer directly to the source of the data reported.

(1) A. K. Huntington, "The Effect of Temperatures Higher than Atmospheric on Tensile Tests of Copper and its Alloys and a Comparison with Wrought Iron and Steel," *Journal*, Inst. Metals, Vol. 8, p. 126 (1912).

(2) G. D. Bengough and D. Hanson, "The Tensile Properties of Copper at High Temperatures," *Journal*, Inst. Metals, Vol. 12, p. 56 (1914).

(3) Clair Upthegrove and A. E. White, "Available Data on the Properties of Non-Ferrous Metals and Alloys at Various Temperatures," *Proceedings*, Am. Soc. Testing Mats., Vol. 24, Part II, p. 88 (1924).

(4) "A Bibliography on Effect of Temperature on Properties of Metals," ASME Research Publication, New York, N.Y. (1928).

(5) Jerome Strauss, "Metals and Alloys for Industrial Applications Requiring Extreme Stability," *Transactions*, Am. Soc. Steel Treating, Vol. 16, p. 191 (1929).

(6) W. B. Price, "Properties of Copper and Some of its Important Industrial Alloys at Elevated Temperatures," Symposium on Effect of Temperatures on the Properties of Metals, ASME-ASTM Joint Publication, p. 340 (1931). (Symposium issued as separate publication, *STP No. 12*.)

(7) W. H. Bassett, Discussion of W. B. Price's paper. Symposium of Effect of Temperature on the Properties of Metals, ASME-ASTM Joint Publication, p. 351 (1931). (Symposium issued as separate publication, *STP No. 12*).

(8) A. Krupkowksi, "Propriétés Méchanique du Cuivre," *Revue de Métallurgie*, Vol. 28, pp. 641–652 (1931); also, Vol. 29, pp. 74–92 (1932).

(9) F. Pester, "Festigkeitsprüfungen an Stangen und Drahten bei tiefen Temperaturen," *Zeitschrift für Metallkunde*, Vol. 24, pp. 67–72 and 115–120 (1932).

(10) M. Kuroda, "Tensile Properties of Copper at Low Temperatures," *Scientific Papers*, Inst. Physical, and Chemical Research, Tokyo, Vol. 19, pp. 163–167 (1932).

(11) E. W. Colbeck and W. E. MacGillivray, "The Mechanical Properties of Metals at Low Temperatures, Part II—Non-Ferrous Metals," *Transactions*, Inst. Chem. Engrs., London, Vol. II, pp. 107–122 (1933).

(12) W. Broniewski and K. Wesolowski, "L'influence de là Température sur les Propriétés Méchaniques du Laitons," *Revue de Métallurgie*, Vol. 30, pp. 396–401 and 451–457 (1933).

(13) W. J. De Haas and Sir R. Hadfield, "On the Effect of Temperature of Liquid Hydrogen (—252.8°C) on the Tensile Properties of Forty-One Specimens of Metals," *Philosophical Transactions*, Royal Soc., (London), Vol. A-232, pp. 297–332 (1933).

(14) C. S. Smith, "Mechanical Properties of Copper and Its Alloys at Low Temperatures: a Review," *Proceedings*, Am. Soc. Testing Mats., Vol. 39, p. 642 (1939).

(15) A. Pomp, A. Kirsch and G. Haupt, "Kerbschlagzähigkeit Legierter Stahle bei Temperaturen von +20° bis —253°C.," *Mitteilungen aus dem Kaiser Wilhelm Institut für Eisenforschung*, Düsseldorf, Vol. 21, pp. 219–230 (1939).

(16) A. Pomp, A. Kirsch and G. Haupt, "Härteprüfung und Zerreisversuche an legierten Stahlen bei tiefen Temperaturen," *Ibid.*, pp. 231–241.

(17) C. S. Smith, "Proportional Limit Tests on Copper Alloys," *Proceedings*, Am. Soc. Testing Mats., Vol. 40, p. 864 (1940).

(18) P. G. Jones and H. F. Moore, "An Investigation of the Effect of Rate of Strain on the Results of Tension Tests of Metals," *Proceedings*, Am. Soc. Testing Mats., Vol. 40, p. 610 (1940).

(19) M. J. Manjoine and A. Nadai, "High Speed Tension Tests at Elevated Temperatures," *Proceedings*, Am. Soc. Testing Mats., Vol. 40, p. 822 (1940).

(20) C. S. Smith and R. W. Van Wagner, "The Tensile Properties of Some Copper Alloys," *Proceedings*, Am. Soc. Testing Mats., Vol. 41, p. 825 (1941).

(21) P. G. Jones and H. F. Moore, "Effect of Rate of Strain on Yield Strength, Tensile Strength, Elongation and Reduction of Area in Tension Tests." *Proceedings*, Am. Soc. Testing Mats., Vol. 41, p. 488 (1941).

(22) A. Nadai and M. J. Manjoine, "High Speed Tension Tests at Elevated Temperatures," Parts II and III, *Journal of Applied Mechanics*, Vol. 8, p. A-77 (1941).

(23) H. W. Gillett, "Impact Resistance and Tensile Properties of Metals at Subatmospheric Temperatures," Project No. 13 of the Joint ASME-ASTM Research Committee on the Effect of Temperature on the Properties of Metals, Am. Soc. Testing Mats., Aug., 1941. (Issued as separate publication, *STP No. 47.*)

(24) J. L. Everhart, W. E. Lindlief, J. Kanegis, P. G. Weissler and F. Siegel, "Mechanical Properties of Metals and Alloys," Circular of the Nat. Bureau Standards C447, Dec. 1943.

(25) D. J. McAdam, Jr. and R. W. Mebs, "The Technical Cohesive Strength and other Mechanical Properties of Metals at Low Temperatures," *Proceedings*, Am. Soc. Testing Mats., Vol. 43, p. 661 (1943).

(26) D. J. McAdam, Jr., G. W. Geil and D. H. Woodard, "Influence of Rate of Strain and Temperature on the Mechanical Properties of Monel Metal and Copper," *Proceedings*, Am. Soc. Testing Mats., Vol. 46, p. 902 (1946).

(27) Copper Development Assn. "Copper and its Alloys in Engineering and Technology," Copper Development Assn. Publication No. 43, p. 14, London (1948).

(28) N. L. Mochel, "Effects of Subatmospheric Temperatures on the Properties of Non-Ferrous Metals," Symposium on Effects of Low Temperatures on the Properties of Materials, Am. Soc. Testing Mats., p. 47 (1950). (Symposium issued as a separate publication, *STP No. 78.*)

(29) R. A. Wilkins and E. S. Bunn, "Copper and Copper Base Alloys," McGraw-Hill Book Company, Inc., New York and London (1943).

(30) W. Köster, "Die Temperaturabhängigkeit des Elastizitätsmoduls reiner Metalle," *Zeitschrift, Metallkunde*, Vol. 39, No. 1, pp. 1–9 (1948).

DISCUSSION

Mr. W. M. Baldwin, Jr.[1]—Information with respect to grain size would be of interest because of its effect on ductility.

May I suggest the possibility that rather than the type of fracture causing a low or high reduction in area, as the case may be, the magnitude of the reduction of area controls or sets the type of fracture.

Mr. J. T. Richards.[2]—The authors have reported that copper strip rolled 5 to 7 per cent has a higher elastic modulus than the annealed material tested. Although there is general agreement that lattice distortion resulting from cold work usually causes a decrease in modulus, the literature seems to be divided on this point in the case of copper.

The accompanying Table II, which summarizes several past investigations, indicates that cold-worked copper generally has the higher modulus. In the case of cold work produced by overstraining in tension, small reductions cause a sharp drop in modulus, followed by gradual recovery with greater reductions (**3**, **8**).[3]

Since Table II shows that modulus of elasticity is apparently a function of annealing temperature, comparisons between annealed and cold-worked material should give some indication of annealing time and temperature. It may also be possible that annealed material may contain minute waves or buckles, which would give reduced values for modulus of elasticity. A similar effect caused by kinks in wire has been previously noted (**4**).

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[1] Research Professor, Metals Research Laboratory, Dept. of Metallurgical Engineering, Case Institute of Technology, Cleveland, Ohio.

[2] Development Engineer, The Beryllium Corp., Reading, Pa.

[3] The boldface numbers in parentheses refer to the list of references appended to this discussion, see p. 1022.

Do the authors have data available regarding the influence of time on modulus and strength values, particularly at elevated temperatures?

MR. A. I. BLANK[4] (*by letter*).—The authors have made a valuable contribution to existing knowledge of the mechanical properties of copper. The results demonstrate the effects of several important variables on those properties. It is of considerable value to have all of this information assembled in a single paper.

TABLE II.—THE INFLUENCE OF COLD WORK ON THE ELASTIC MODULUS OF COPPER.

Reported By	Reference	Form	Condition	Modulus of Elasticity, psi	
				Annealed	Cold Worked
Wertheim	1			14 980 000	17 740 000
Honda and Yamada	2	Rod		17 370 000	16 660 000
Kuntze	3	Rod	Cold Drawn	18 230 000	18 550 000
Batson and Hyde	4	Rod	Rolled		15 000 000
		Wire	Cold Drawn	16 000 000	18 500 000
Phillips and Smith	5	Wire	Cold Drawn		18 000 000
			1 hr. at 400 C	13 400 000	
McKeown and Hudson	6	Rod	Cold Drawn		18 300 000
			½ hr at 650 C	17 900 000	
Smith and Van Wagner	7	Tough Pitch Rod		15 820 000	16 950 000
		Oxygen Free Rod		16 200 000	17 540 000
McAdam and Mebs	8	Rod	Cold Drawn		18 500 000
			22 hr at 600 C	18 200 000	
			5 hr at 800 C	18 900 000	
Gohn and Arnold	9	Strip		17 400 000	16 400 000
Average				16 760 000	17 467 000

Some work done several years ago in the research laboratory of the Chase Brass and Copper Co. confirms the author's findings that the effect of speed of testing on mechanical properties is small for the range of speeds ordinarily used in static tests. Stress-strain tests were made at 400 F on specimens of annealed electrolytic tough pitch copper, cartridge brass, 70 per cent, Naval brass and high-silicon bronze, (A) in a Baldwin-Southwark Universal testing machine of 50,000-lb capacity. Strain rates of 0.01, 0.1, and 0.4 per cent elongation in 2 in. per min were used. The effect of rate of strain on yield strength at 0.5 per cent extension under load was found to be only slight, although in the plastic region the total strain increased for any given stress as the strain rate decreased. The average increase in yield strength at 0.5 per cent extension under load effected by a forty-fold increase in strain rate (from 0.01 to 0.4 per cent elongation in 2 in. per min) was only 4 per cent for the four materials tested. The results are summarized in the following table:

Alloy	Temper	Yield Strength at 0.5 per cent Extension Under Load at Indicated Strain Rate, psi			Difference, per cent
		0.01[a]	0.1[a]	0.4[a]	0.01–0.4
Electrolytic tough pitch copper	0.035 mm average grain size	5 000	5 330	5 450	8
Cartridge brass, 70 per cent	0.035 mm average grain size	15 400	15 400	15 400	0
Naval brass,	Annealed at 1050F for 1 hr	21 800	23 100	23 100	5
High-silicon bronze, (*A*)	0.035 mm average grain size	14 800	14 950	15 250	3

[a] Per cent in 2 in. per min.

MR. W. H. MUNSE (*author's closure*).—Metallurgical studies of the as-received materials were made on longitudinal and transverse sections cut from the edge and center of the parent plates. Typical micro-structures of several of these sections are presented in Fig. 14.

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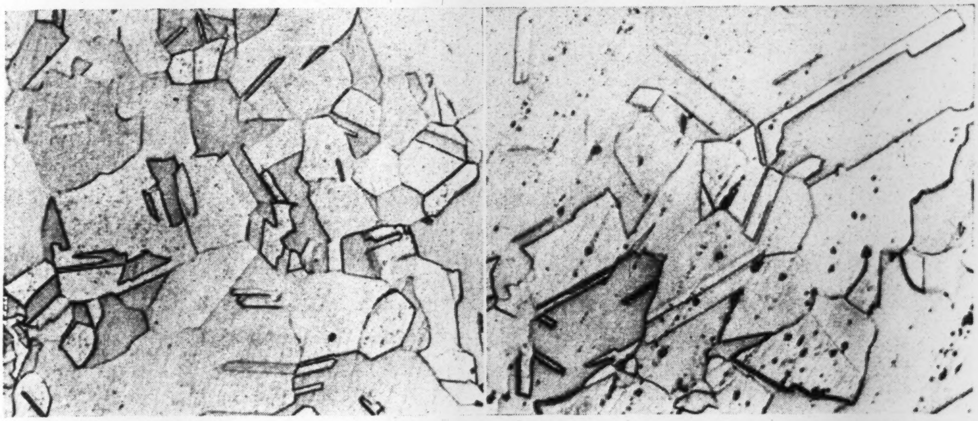
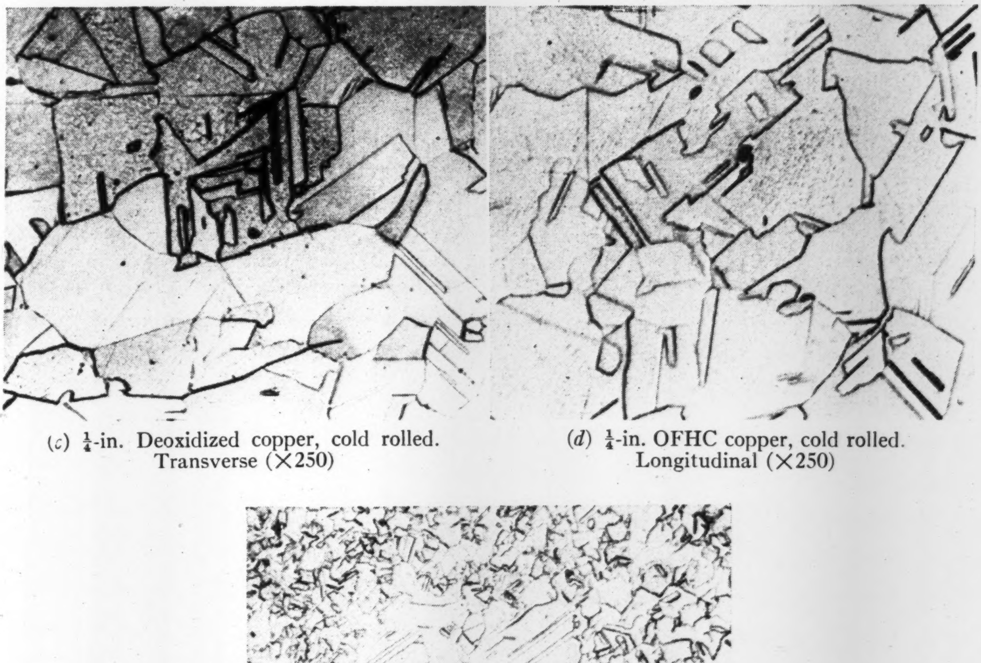
[4] Assistant Research Metallurgist, Chase Brass and Copper Co., Waterbury, Conn.



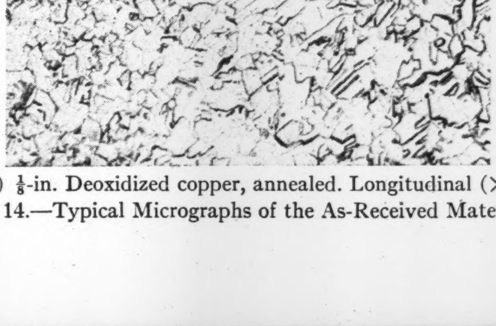
(*a*) $\frac{1}{8}$-in. OFHC copper, annealed. Longitudinal (×250)

(*b*) $\frac{1}{4}$-in. electrolytic copper, cold rolled. Longitudinal (×250)



(*c*) $\frac{1}{4}$-in. Deoxidized copper, cold rolled. Transverse (×250)

(*d*) $\frac{1}{4}$-in. OFHC copper, cold rolled. Longitudinal (×250)



(*e*) $\frac{1}{8}$-in. Deoxidized copper, annealed. Longitudinal (×50)

FIG. 14.—Typical Micrographs of the As-Received Material.

TABLE III.—GRAIN SIZES OF AS-RECEIVED MATERIAL.

Temper	Thickness of Material, in.	Type of Copper	Longitudinal Section Grain Size, mm		Transverse Section Grain Size, mm, Center of Plate
			Edges of Plate	Center of Plate	
Annealed	⅛	Electrolytic	0.035	0.040	0.040
		Deoxidized	0.045	0.045	0.045
		O.F.H.C.	0.045	0.045	0.045
	¼	Electrolytic	0.045	0.045	
		Deoxidized	0.043	0.043	
		O.F.H.C.	0.048	0.048	
Cold-rolled	⅛	Electrolytic	0.038	0.042	
		Deoxidized	0.045	0.047	
		O.F.H.C.	0.043	0.040	
	¼	Electrolytic	0.050	0.050	0.048
		Deoxidized	0.045	0.045	0.045
		O.F.H.C.	0.045	0.045	0.045

The micrographs of the electrolytic tough pitch copper revealed small stringers of Cu_2O elongated in the direction of rolling. Similar micrographs of the deoxidized copper showed tiny stringers of the products of the deoxidation reaction, also distributed in the direction of rolling. No inclusions, however, were noted in the oxygen-free high-conductivity copper.

The specimens showed excellent uniformity of grain size in all of the plates except the ⅛-in. sheets of annealed deoxidized high-phosphorus copper which contained a band of coarse grains through the middle as shown in Fig. 14 (*e*). This irregularity, however, was not noted in the ¼-in. plate of the same base material. The grain size measurements of the specimens cut from the center and edge of the as-received plates are given in Table III.

It is of interest to note the number of references offered by Mr. Richards which indicate, as do the tests, reported in this paper, that cold-rolled copper generally has a higher modulus of elasticity than the annealed copper. A number of the results reported by Mr. Richards appear to be for materials which were annealed after rolling or drawing. The tests reported by the authors show the same general results for materials which were annealed at 1150 F for one-half hour as compared with cold-rolled material which had been annealed at 1150 F for one-half hour prior to rolling.

The authors would like to express their appreciation to the discussers for their comments and for the additional information and references which they have provided.

REFERENCES

(1) G. Wertheim, *Pogg. Ann.*, Vol. 78, p. 391 (1849).

(2) K. Honda and R. Yamada, "On the Change of Elastic Constant in Metals Caused by Cold-Working," Science Reports of the Tohoku Imperial University, Vol. 17, pp. 723–742 (1928).

(3) W. Kuntze, "The Relationship between the Elastic Strain Coefficient Alpha and the Previous Treatment of Copper," *Zeitschrift für Metallkunde*, Vol. 20, April, 1928, pp. 145–150.

(4) R. G. Batson and J. H. Hyde, "Mechanical Testing," Vol. I, Testing Materials of Construction, Chapman and Hall, Ltd., London (1922).

(5) A. J. Phillips and A. A. Smith, Jr., "Effect of Time on Tensile Properties of Hard-Drawn Copper Wire," *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part II, pp. 263–275 (1936).

(6) J. McKeown and O. F. Hudson, "Stress-Strain Characteristics of Copper, Silver and Gold," *Journal*, Inst. Metals (London), Vol. 60, pp. 571–592 (1937).

(7) C. S. Smith and R. W. Van Wagner "The Tensile Properties of Some Copper Alloys," *Proceedings*, Am. Soc. Testing Mats., Vol. 41, pp. 825–845 (1941).

(8) D. J. McAdam and R. W. Mebs, "Tensile Elastic Properties of Nickel, Copper, Open-Hearth Iron, and Typical Steels," *Journal of Research*, National Bureau of Standards, Vol. 28, March, 1942, pp. 311–378.

(9) G. R. Gohn and S. M. Arnold, "The Stress-Corrosion Properties of Some Non-Ferrous Sheet Metals," Symposium on Stress-Corrosion Cracking of Metals, Published Jointly by Am. Soc. Testing Mats. and Inst. Metals Div., Amer. Inst. Mining and Metallurgical Engrs., pp. 155–167 (1944). (Issued as separate publication, *STP No. 64*).

RHEOTROPIC BRITTLENESS: GENERAL BEHAVIORS[*][1]

By E. J. Ripling and W. M. Baldwin, Jr.[2]

Synopsis

The brittle behavior of a metal with a hexagonal type crystal structure (pure zinc) at temperatures less than the transition value was found to be largely strain curable (rheotropic). This suggested that rheotropism, heretofore reported only in steels, is a general property of materials that show a transition temperature.

Nor is rheotropism confined to the brittleness induced by low temperatures. The present report shows the low ductility of zinc at high strain rates to be rheotropic. Since the brittleness of notched steels was recently found to be rheotropic it can be assumed that the brittleness induced by any of the three known embrittling variables (temperature, strain rate, stress state) is strain sensitive.

Prior Considerations

The ductility of a steel will drop as its temperature is lowered, and not infrequently it will drop sharply within a narrow range of temperatures (called the transition temperature range) as shown by curve A in Fig. 1.

Interesting results are obtained when the steel is first stretched an arbitrary amount at a temperature where the ductility is normally high (this deformation being usually termed a prestrain) and is then stretched to failure after the specimen has been cooled to a lower temperature. So long as the lower testing temperature is still above the transition temperature, the ductility is decreased by prestrain as shown in curve C in Fig. 1. If the test temperature is below the transition temperature, however, the sharply reduced ductility of the original unworked steel is actually improved by prestrain as shown in curve D in Fig. 1. Extrapolation of the portion dd of the curve D (the so-called strain-stabilized portion of the curve) back to zero prestrain and an extrapolation of the super-transition-temperature branch of the ductility *versus* temperature curve (section a-a) to sub-transition temperatures, leads to the same value of ductility (point x in Fig. 1). This behavior indicates that the reduced ductility of unworked steels below the transition temperature is due to some impediment operating in the steel at sub-transition temperaturers which prestrain at higher temperatures neutralizes. The reduction in ductility from the extrapolated value x in Fig. 1 to the actual value of y—since it is responsive to flow—is termed rheotropic[3] brittleness (**1**).[4]

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[*] Presented at the Fifty-fourth Annual Meeting of the Society, June 18–22, 1951.

[1] This paper is based on a report of a research program conducted at the Metals Research Laboratory, Department of Metallurgical Engineering, Case Institute of Technology in cooperation with the Office of Naval Research, U. S. Navy.

[2] Senior Research Associate, and Research Professor, respectively, Metals Research Laboratory, Department of Metallurgical Engineering, Case Institute of Technology, Cleveland, Ohio.

[3] This word is compounded from the Greek. "Tropism" is borrowed from the biologist who uses it to mean "the innate tendency of an organism to react in a definite manner to external stimuli." Phototropism is an example. In the present case, the external stimulus is flow for which the Greek word "rheos" has been used so much of late.

[4] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1030.

This behavior—described in such narrow terms above—stimulates interest. In the first place, how general is the behavior? For example, metals other than steels are known to show sharp drops in ductility with decreasing temperature. Is the sharp ductility drop in these metals rheotropic? Or, for example, the ductility drop found in steels (and other metals) occurs not only when temperature is decreased, but also when either strain rate or hydrostatic tension are increased. Will prestrain at low strain rates or low hydrostatic tensions regenerate the ductility denied to the metal at high strain rates or high hydrostatic tensions?[5]

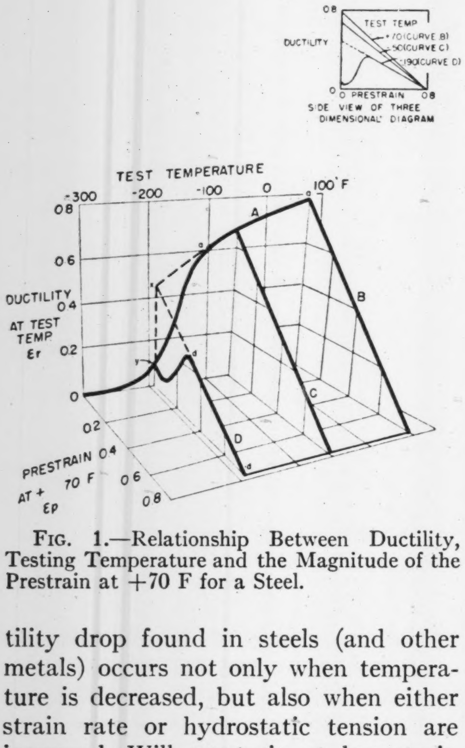

FIG. 1.—Relationship Between Ductility, Testing Temperature and the Magnitude of the Prestrain at +70 F for a Steel.

In the second place, what is the mechanism by which prestrain neutralizes the rheotropic impediment? The embrittlement of metals at low temperatures or high strain rates or high hydrostatic tension is a complete mystery, and knowledge of the reaction of the embrittlement to any stimulus will serve to help solve that mystery. Is the restoration of ductility due merely to a realignment of microcracks to a more favorable orientation during prestraining? Or does prestrain at high temperatures nucleate slips on planes where nucleation normally cannot occur at sub-transition temperatures?

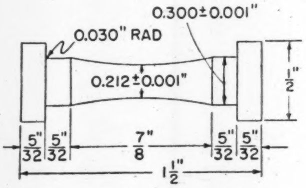

FIG. 2.—Test Specimen.

The present investigation was designed to provide some answer to these questions. A number of critical experiments were carried out with zinc, each designed to reveal some information of a basic nature about rheotropic brittleness. Zinc was chosen because it possesses a high transition temperature and can be obtained in high purities.

MATERIAL AND EQUIPMENT

The zinc used in this investigation was obtained as ¾-in. diameter extruded rods of 99.99 per cent purity. Specimens of the type shown in Fig. 2 were machined from these rods, after which the specimens were annealed at 475 F for 1 hr and air-cooled in order to eliminate any cold work that took place in the machining operation.

The tensile strains (at 0.05, 1.0 and 10 in. per min) were made with the aid of equipment previously described (**4**). In order to obtain strains at temperatures between room temperature and +212 F, warm water was used with this equipment in place of a coolant. Higher temperatures were obtained with the use of hot oil.

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[5] One set of data (**1**) indicates that prestrain at low hydrostatic tension does, in fact, restore ductility lost under high hydrostatic tension.

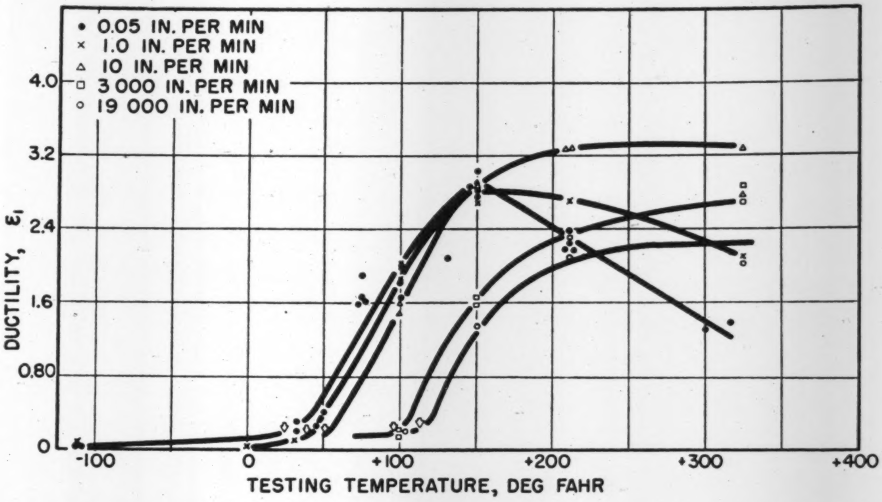


FIG. 3.—Effect of Testing Temperature on the Ductility at Strain Rates Between 0.05 and 19,000 in. per min.

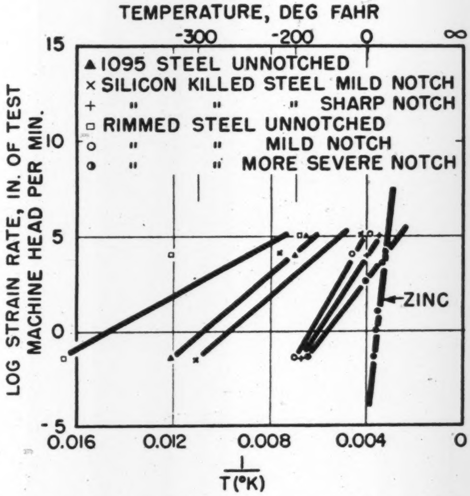


FIG. 4.—Re-Plot of Some Steel Data (from Jones and Worley (3)) and for the Zinc Data Shown in Fig. 3 to an Arrhenius Type Plot. Heel of the transition temperature curve is taken as the transition temperature value.

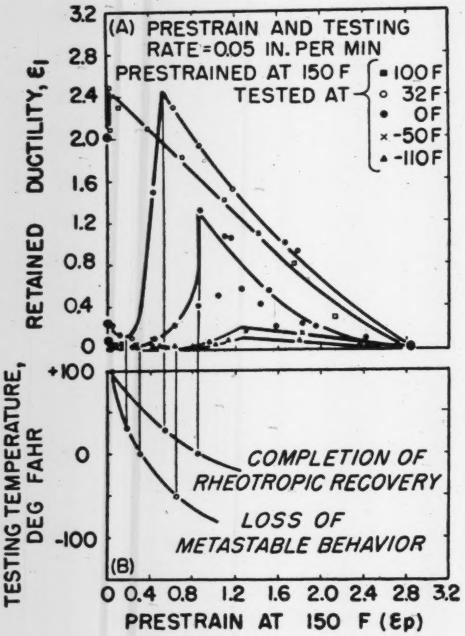
The strains at 3000 and 19,000 in. per min were made by adapting a drop hammer for tensile strains.

Ductility measurements were made by determining the specimen diameters on a microcomparitor before and after each straining operation.

PRESENT STUDIES

Figure 3 is a plot of ductility (ε = ln of the ratio of the original cross-sectional area of the specimen to the cross-sectional area at fracture) as a function of temperature for 99.99 per cent zinc. The sharp ductility drop with decreasing temperature is at once apparent. The transition temperature is also seen to be clearly a function of strain rate, moving to higher values as the strain rate is increased. The temperature and strain rate corresponding to the heel of each curve (see arrows in Fig. 3) are plotted to Arrhenius coordinates (log rate *versus* reciprocal absolute temperature) in Fig. 4. A plot of this type was used by Zener and Holloman to show the relationship between testing temperature and strain rate in determining flow properties (6) and subsequently modified by Morkovin (7) to apply to fracturing as was the case here. The data in Fig. 4 adhere to a straight line. The slope of the line is quite steep and the line would not appear to extrapolate to the same approximate values of strain rate at ($1/T = 0$) as do the lines for steels.[6] Yet it is felt that the data are correct since transition temperatures for zinc determined by impact tests do not differ significantly from those determined by tension tests indicating a low sensitivity of the transition temperature in the case of zinc to strain rate (and notch effects) (2).

The ductility values in Fig. 3 drop with increasing temperature (at temperatures higher than the transition value) when the testing is done at low strain rates (0.05 in. per min and 1.0 in. per min). For the interim—until further facts are brought out in this paper—this embrittlement will merely be referred to as a high-temperature embrittlement.



(*B*) Position of the Retained Ductility—Prestrain Maximum and Minimum at Various Testing Temperatures.

FIG. 5.—Ductility at a Variety of Temperatures.

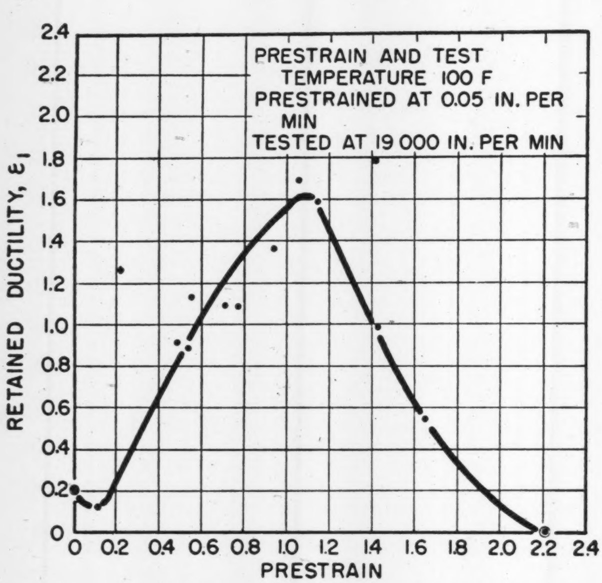


FIG. 6.—Effect of Prestraining Pure Zinc at a Strain Rate of 0.05 in. per min at 100 F on the Ductility Retained at a Strain Rate of 19,000 in. per min at 100 F.

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[6] Many quantities which may be plotted to Arrhenius coordinates yield families of straight lines (each line corresponding to a single material or system) which converge at ($1/T = 0$) to a narrow range of values, that is, dissociation pressures of oxides, diffusion coefficients of metal systems, etc.

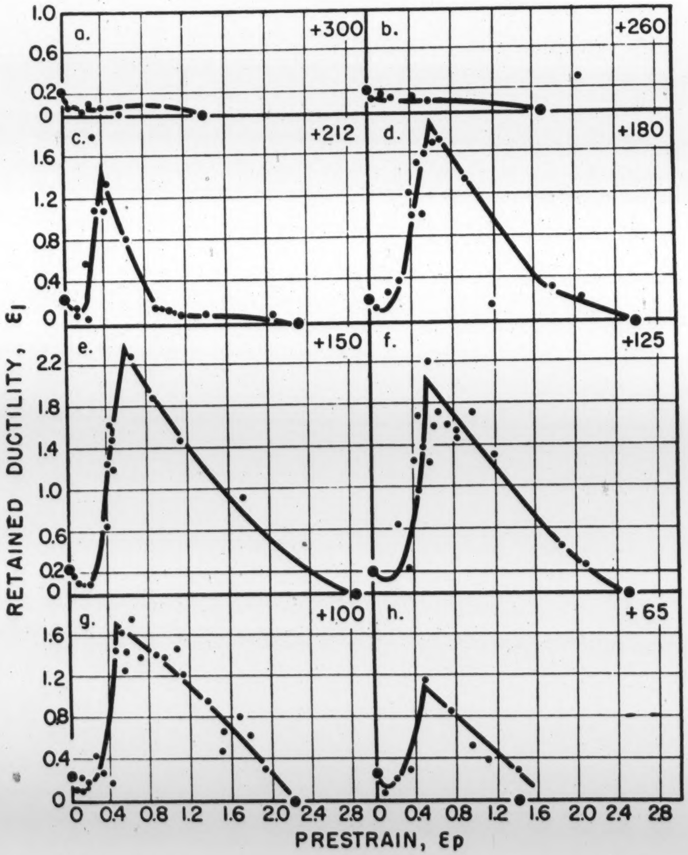

FIG. 7.—Effect of Prestraining at the Temperature Shown on the Ductility at 32 F. (Prestraining and Testing Both at a Strain Rate of 0.05 in. per min.)

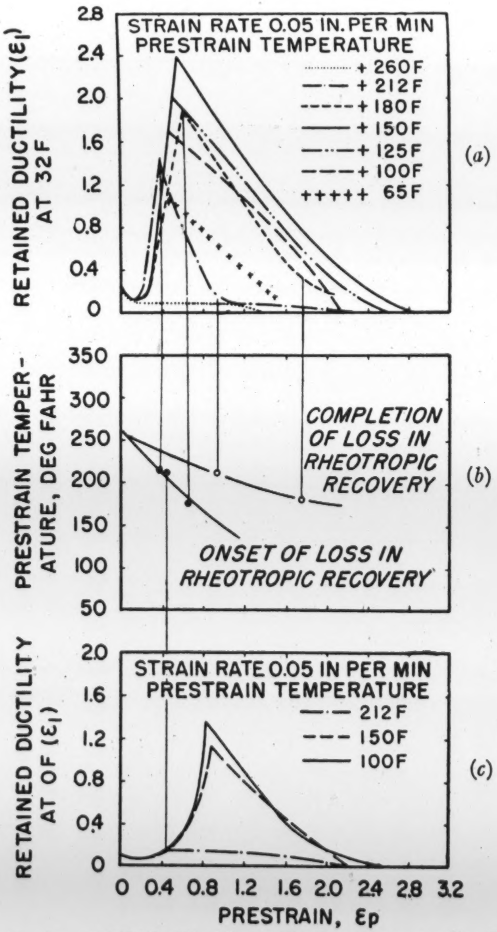

FIG. 8.—Strains at Which Working Effects Obliterate the Rheotropic Recovery at Various Prestraining Temperatures.

Experiments in which zinc was prestrained at a rate of 0.05 in. per min at 150 F and then tested to fracture at lower temperatures (100 F, 32 F, 0 F, −50 F, and −110 F) are reported in Fig. 5(*A*). It is clear that prestraining effects the same type of improvement in ductility in zinc as in steel. The sharp ductility drop as testing temperatures are decreased for a metal other than steel appears to be rheotropic, and the problem takes on a general rather than a specific interest.

Experiments in which zinc was prestrained at 100 F at the low strain rate of 0.05 in. per min and tested at high strain rates (3000 in. per min) at the same temperature are reported in Fig. 6. Again it is clear that prestraining effects an improvement in ductility. The sharp ductility drop as a function of any of the three embrittling parameters (decreasing temperature, increasing hydrostatic tension, and increasing strain rate) appears to be rheotropic. The problem is quite general.

Zinc was prestrained at a variety of temperatures and tested at 32 F. The ductility retained at this testing temperature as a function of prestrain is given graphically in Fig. 7.

It will be seen that at the given testing temperature the early drop and increase in the different curves practically coincide irrespective of the prestraining temperature, although the strain-stabilized portions of the curves depend upon prestraining temperature. Prestraining the metal at high temperatures (150 F and higher) is not completely effective in regenerating ductility at sub-transition temperatures. In fact, the retained ductility at 32 F of zinc prestrained (at 0.05 in. per min) at 300 F and 260 F never shows any increase. The retained ductility at 32 F of zinc prestrained (at 0.05 in. per min) at 212 F does show an increase with prestrain, but before this increase can reach as high a value as it does with lower prestraining temperatures, some effect associated with the high-temperature embrittlement sets in at a strain of about 0.4, depressing the retained ductility value until, at a prestrain of about 0.9, the improvement in retained ductility is completely lost.

Figure 8 is a graph showing the prestrains at which the beginning and completion of loss in improvement in retained ductility occurs for the various prestraining temperatures studies. The lower the temperature the greater is the prestrain at which the associated effect sets in and destroys rheotropic recovery. In order to determine whether or not the lowered ductilities at high prestrains were a result of an aging phenomenon that took place between the prestraining and testing, specimens were prestrained at +150 and +212 F and then tested at +32 F after 10 min. and 4 days. The test results obtained after these two aging intervals were the same, indicating that the factor that causes the suppressed ductility was operating during the test.

As may be seen in Figs. 7 and 8, the early portions of the retained ductility *versus* prestrain curves for different prestrain temperatures are virtually coincident at a constant testing temperature. They vary, however, as a function of test temperature. In Fig. 5(*B*) are plotted the prestrains at which the minimum and maximum of the retained ductility *versus* prestrain curves occur as a function of test temperature. As test temperature descends, these prestrains become larger.

It is difficult to determine the hypothetical values of ductility at sub-transition temperatures free from the effects of impediment (as was done for example in Fig. 1 by extrapolating the strain-stabilized portion of the retained ductility *versus* prestrain curve back to zero prestrain) since a second factor interfered with prestraining at low strain rates at super-transition temperatures.[7]

The data for prestraining at 150 F and testing at lower temperatures allow a rough estimation of the hypothetical values. The strain stabilized portions of the curves of Fig. 5(*A*) were extrapolated to zero prestrain, and these values are entered in Fig. 9 (as open circles). The values are seen to extend the super-transition portion of the ductility *versus* temperature curve free from effects of hot work so that the super-transition temperature curve for the low strain rates forms a family of curves with the high strain rate curves shown in Fig. 3. It is seen that the impediment-free curve has a downward trend with decreasing temperature. This trend was also noted in the case of a $2\frac{3}{4}$ per cent silicon transformer steel (1).

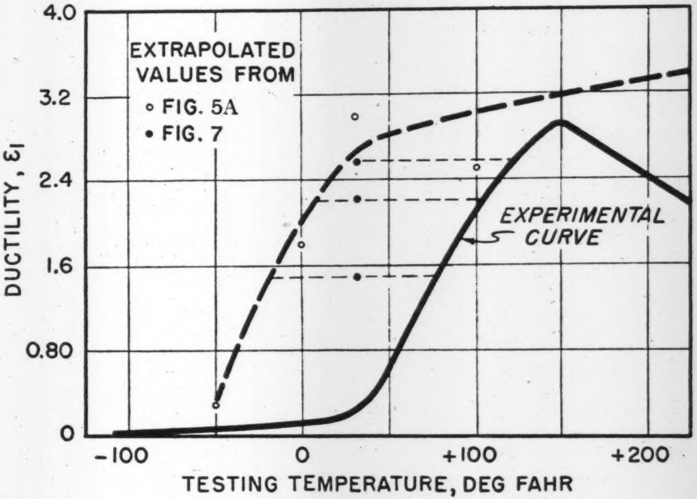

FIG. 9.—Actual Ductility of Zinc and Suggested Ductility in the Absence of Rheotropic Embrittlement.

It was thought interesting to inquire into the effects of prestraining at temperatures in the transition temperature range on sub-transition ductilities. The data in Figs. 7 (*f*, *g*, and *h*) indicate that rheotropic recovery is achieved in these cases, although the hypothetical value of retained ductility at zero prestrain (obtained by extrapolating the strain stabilized portion of the curves backward) never exceeds the actual ductility value of the prestraining tempueratre. These values are entered in Fig. 9 also.

At this point the general features of the high-temperature embrittlement can be pointed out. At low strain rates and high temperatures, the ductility was found to decrease with increasing testing temperature. This incidentally, is in agreement with some data presented by Jeffries (5) on annealed copper wire which was found to exhibit a similar high-temperature embrittlement at low strain rates. When specimens were prestrained in the temperature range of this high-temperature embrittlement, the improvement in low-temperature properties was not realized over the complete range of prestrains. As a matter of fact, the higher the prestraining temperature the more narrow is the prestrain region over which low temperature performance was improved. This behavior would lead one to suspect that the zinc was being hot-worked at super-transition temperatures. However, a microscopic examination showed some recrystallization to have taken place in all the specimens (even those strained at high strain rates).

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[7] Prestraining at high strain rates at super-transition temperatures (where high-temperature embrittlement doesn't interfere) present many experimental difficulties.

Acknowledgments:

This investigation was conducted in cooperation with the Office of Naval Research, U. S. Navy, to whom the authors owe a debt of gratitude for permission to publish this work.

The kind assistance given by Mr. L. J. Ebert in preparing the text, as well as the help of Messrs. Howe and Mayersky who conducted many of the tests, and the General Electric Co., which supplied the liquid nitrogen used in this study, are also gratefully acknowledged.

REFERENCES

(1) E. J. Ripling and W. M. Baldwin, Jr., "Rheotropic Embrittlement of Steel," *Transactions*, Am. Soc. Metals, Vol. 43, pp. 778–810 (1951).

(2) F. Erdmann-Jesnitzer and W. Hofmann, "Some Observations on the Impact Bending Strength and Notch Bar Toughness of Zinc Alloys," *Zeitschrift für Metallkunde*, Vol. 35, October, 1943, pp. 211–212.

(3) P. G. Jones and W. J. Worley, "An Experimental Study of the Influence of Various Factors on the Mode of Fracture of Metals," *Proceedings*, Am. Soc. Testing Mats., Vol. 48, p. 643 (1948).

(4) E. J. Ripling and G. Tuer, "An Apparatus for Low Temperature Tensile Testing," *Product Engineering*, Vol. 20, No. 1, p. 103 (1949).

(5) Z. Jeffries, "Effect of Temperature, Deformation, and Grain Size on the Mechanical Properties of Metals," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 60, p. 474 (1919).

(6) C. Zener and J. H. Holloman, "Plastic Flow and Rupture of Metals," *Transactions*, Am. Soc. Metals, Vol. 33, pp. 163–215 (1944).

(7) D. Morkovin, Discussion of the paper by C. Zener and J. H. Holloman (6), pp. 221–226.

DISCUSSION

Mr. W. A. Reich.[1]—Several years ago in working with zinc it was noticed that a specimen of annealed zinc put through the rolling mill would not take a heavy draft and the operator commented, "You've got to hit it easy first and then it will take quite a bit."

Taking a slight reduction first allowed further working of the material quite nicely without excessive edge cracking.

At that time this was explained on the basis of zinc's being hexagonal in crystal structure and having one slip system along the basal plane. The small reduction induced twinning which gave another effective slip system at right angles to the original, and thus the material behaved in a more ductile fashion. Have the authors studied this possibility in connection with this project as applying to the hexagonal metals?

Mr. J. H. Westbrook[2] (*presented in written form*).—This paper not only provides the first demonstration of rheotropism in a non-ferrous material but also presents the results of a wider exploration of the effects of the rheotropic variables (test temperature and strain rate, prestrain temperature and strain rate, and prestrain itself) than has heretofore been available for any material. It is to be hoped that further exhaustive experimental studies will soon be forthcoming for other materials and thus permit explanation of the mechanism by which "prestrain neutralizes the rheotropic impediment."

It is believed that the data presented are amenable to further analysis. Another picture of the effects of prestrain on the ductility than that presented by the authors can be achieved by replotting the data of the authors' Fig. 5 in the more familiar ductility-temperature plot. The usefulness of this type of plot has been brought out by the writer's associate, Mr. H. C. Rogers, who has reinterpreted the bulk of the existing literature on rheotropic behavior from this viewpoint (to be published). Such a plot for the data of the present paper is shown in Fig. 10. While it is realized that the data are not as extensive as might be desired for this type of analysis, it will be observed that by prestraining at 150 F the brittle-ductile transition temperature may be lowered from 78 F to −5 F. Further, the maximum lowering of the transition temperature is reached with a prestrain of 1.2, either higher or lower prestrains giving higher transition temperatures. This not only provides a further significant observation which must be incorporated into any mechanistic hypothesis, but also raises the practical question—would prestrains induced at other temperatures or strain rates give further reductions in the transition temperature?

The authors observe that prestraining the metal at temperatures above 150 F is not completely effective in regenerating ductility at sub-transition temperatures. It is suggested that this is probably true only for a limited range of prestraining strain rates, and that at higher strain rates of prestrain, the regenerative effect on sub-transition ductility might appear even with the higher prestrain temperatures.

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Figure 8(*b*) is incomplete and by virtue of its incompleteness rather misleading. A complete plot made up from the data of Figs. 7 and 8(*c*) is shown in Fig. 11. The portions of the curves in Fig. 11 lying above about 150 F, that is the only data plotted by the authors, are of little significance since these lie in the region of "high-temperature brittleness." The lower portions of these same curves, however, are significant and show that the prestrain required for maximum ductility decreases slightly with decreasing prestrain temperature, and that it is less for a test temperature of 32 F than for 0 F.

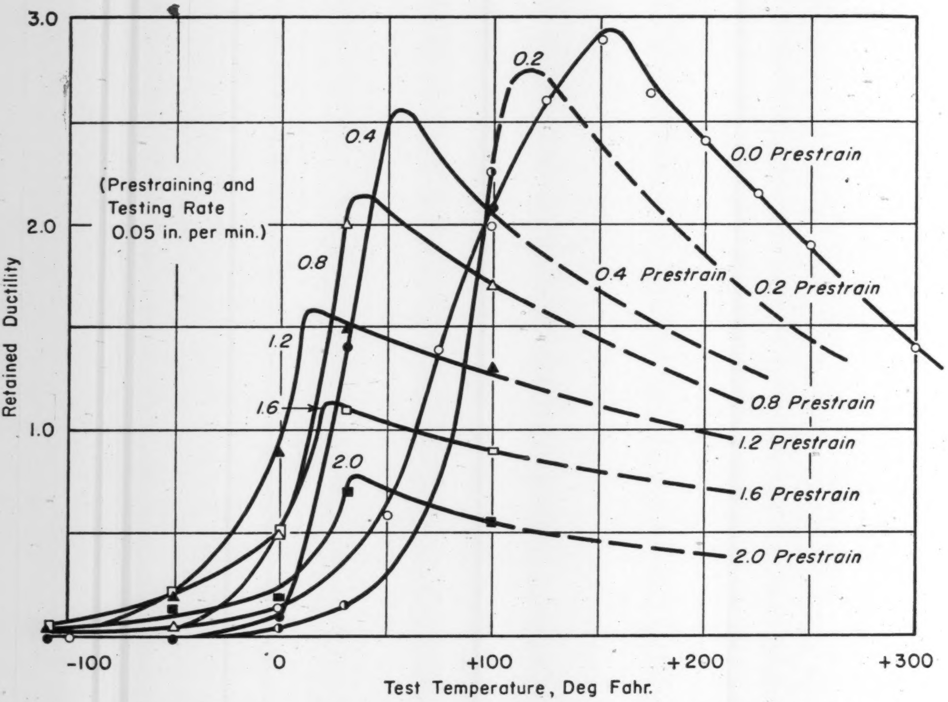

Fig. 10 Effect of Testing Temperature on Ductility After Prestraining Various Amounts at 150 F.

The authors' usage of such phrases as "loss of rheotropic recovery" and "destruction of rheotropic recovery" is somewhat regrettable, particularly in view of their very lucid presentation of the rheotropic embrittlement concept in their earlier paper (**1**).[3] There they went to some length to show that the typical curve of retained ductility *versus* prestrain for a material exhibiting rheotropic embrittlement may be considered as consisting of three parts. The early portion of such a curve, representing the metastable behavior of the material while under the rheotropic impediment, is separated by a transition region from the latter portion of the curve which represents the stable condition produced by the neutralizing effect of the prestrain on the impediment. Thus it would seem more logical to speak of the point

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[3] The boldface numbers in parentheses refer to the list of references appended to the paper, see p. 1030.

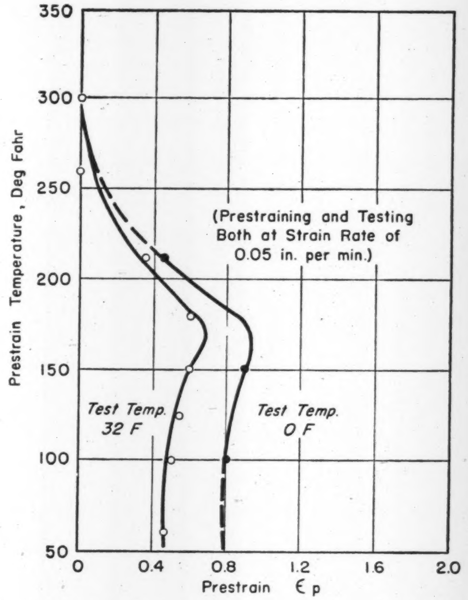

Fig. 11—Prestrain for Maximum Ductility as a Function of Prestrain Temperature.

of complete stabilization rather than that of the onset of loss of rheotropic recovery.

The authors dismiss the possibility of an aging effect to account for low ductilities at high prestrains after obtaining negative results with a few aging experiments. It is not clear from the paper whether the aging in these experiments was carried out at the prestrain temperature, room temperature, or the testing temperature.

In Fig. 9 the authors plot a curve for the suggested ductility in the absence of rheotropic embrittlement from the data of Fig. 5 extrapolated to zero prestrain. They then state that this resultant plot causes the low strain rate curve to become one of a family with those of the higher strain rates as shown in Fig. 3. Such is not the case. If a curve is drawn *through* the extrapolated points, it will be seen to be identical in shape to that

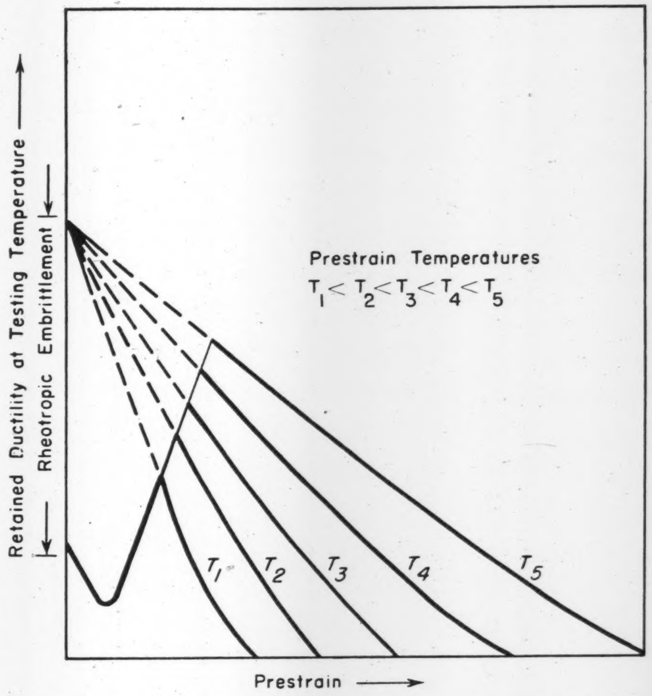

Fig. 12—Effect of Prestrain at Various Temperature on Ductility Lost by Rheotropic Embrittlement.

for the experimental curve for 0.05 in. per min strain rate and merely displaced to lower temperatures. The authors made similar extrapolations of their data from Fig. 7, entered these points also on Fig. 9, and noted that the ductilities thus obtained *never* exceeded those of the experimental curve for temperatures corresponding to the respective prestrain temperatures. Why would any other result be expected? The remarkable thing is that for this case (tested at 32 F) the extrapolated values are identical, within experimental error, with those of the experimental curve. It is suspected that this is merely coincidental.

There seem to be two possibilities. In the simpler case one would suppose that all the curves of Fig. 7 should extrapolate to the same ductility value at zero prestrain since this is the hypothetical ductility at the test temperature. An idealized representation is shown in Fig. 12. The metastable and transition regions of the various curves may well coincide as indicated by the authors' data yet still give different curves in the strain-stabilized region due to the fact that the ductility of the material varies with the prestraining temperature as shown by the intercepts on the horizontal axis. The authors' failure to find a common intercept on the vertical axis might be laid to the fewness of the data, the unavoidable length of the extrapolation, as well as to the appearance of a second phenomenon, "high-temperature embrittlement."

On the other hand, it is possible that for some obscure reason the extrapolated points do not necessarily coincide but rather give a sequence of ductility values proportional to the prestraining temperature, much as the authors indicate in Fig. 9. In this case, it would seem that a whole family of curves like that for 150 F prestrain derived from Fig. 5 and lying between this and the experimental curve might be obtained for various prestrain temperatures. We have attempted to extrapolate the curves for tests at 0 F from the authors' Fig. 8(*c*) and thus obtain other points which would aid either in determining a family of curves as just suggested, or a single extrapolated ductility as suggested above. Unfortunately, the data are too few to permit a clean-cut determination in either case. We, therefore, ask whether the authors have additional data which might be decisive with respect to these two interpretations.

The drop in the ductility curves at high temperatures with low strain rates is designated by the authors as "high-temperature embrittlement" and they cite a single paper (5) describing similar results with copper. This effect is much more widely observed than this brief treatment would imply. In particular, it has been noted also by Oberhoffer for mild steel[4] and by Sauerwald and Wieland for alpha brasses[5]. Recent unpublished research by the writer indicates that this is probably a general effect for all metals and, for ordinary strain rates, occurs always very near one-half the absolute melting point (K). For the case of zinc in the present paper, the authors found high temperature embrittlement setting in at 150 F (339 K). One-half the absolute melting point of zinc is (346 K).

Finally, it would be of interest to know what observations, if any, the authors made of the character of the fracture under the various conditions employed in their experiments.

MR. E. J. RIPLING (*author*).—In reply to Mr. Reich's comment, we have not examined these stretched specimens metallographically or crystallographically very extensively. One of the reasons for this is the fact that the behaviors found to exist were thought to be quite general in that they have been found in a large number of metals. As a matter of fact, it was first uncovered in steels, Mr. Reich's comment is well taken and I think it would probably pay us to look into these factors.

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[4] Doan and Mahla, "Principles of Physical Metallurgy," McGraw-Hill Book Co., New York, N. Y., p. 130 (1941).

[5] *Ibid.*, p. 360.

Mr. Westbrook's comments are much appreciated by the authors. Figure 10 is not unlike a plot previously presented by the authors on a steel (1, Fig. 14), in which it was suggested that prestraining lowers the transition temperature. The material prestrained to an ε_p of 1.2 exhibits the very lowest transition temperature since this is the lowest prestrain investigated by Mr. Westbrook for which the zinc tested at 0 F is on the strain-stable branch of the retained ductility - prestrain curve. The authors prefer to attach no special significance to this point since in rating the worked zinc by the test used here, a prestrain of 1.2 seems optimum. If however, the tests had been of some different type (notched tests, for example) the shape of the metastable and transition branches of the curves would probably be changed and as a consequence a prestrain of 1.2 would no longer be most effective in lowering the transition temperature.

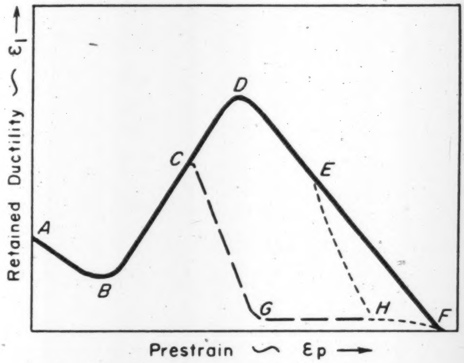

Fig. 13—Suppression of Rheotropic Recovery by the High Temperature Embrittlement.

In the third paragraph of his discussion Mr. Westbrook suggests that the failure of prestrain at super transition temperatures to regenerate ductility below the transition temperature is confined to low prestrain rates. The authors agree as is suggested in the seventh footnote of their paper and the text to which the footnote applies.

Mr. Westbrook's Fig. 11 and his objection to the phrases, "loss of rheotropic recovery," and, "destruction of the rheotropic recovery," are the result of a misunderstanding of Figs. 5(*b*) and 8(*b*). The curves in Fig. 5(*b*) show the dependence of rheotropic recovery on prestrain and testing temperature. Figure 8(*b*), on the other hand, presents the influence of the high-temperature embrittlement on rheotropic recovery. The manner in which these two phenomena are separated is shown in Fig. 13. The solid curve in Fig. 13 is a schematic retained ductility - prestrain curve in the absence of the high-temperature embrittlement. *AB* is the metastable branch of the curve, *BD* the transition branch, and *DF* the stable branch. Point *D* is the point of complete stabilization. Prestraining in the hot embrittlement region can destroy stabilized behavior as shown by the dotted curve (here *E* represents the onset of loss of rheotropic recovery and *H* the completion of loss of rheotropic behavior) or even prevent attainment of stabilized behavior as shown by the dashed curve (here *C* represents the onset of loss of rheotropic recovery and *G* the completion of loss of rheotropic behavior).

Figure 8(*b*) is a plot of the prestrains at which loss of rheotropic recovery set in and was completed as defined by the paragraph above. The points which Mr. Westbrook has added to Fig. 8(*b*) to produce Fig. 11 are points of complete stabilization again as defined in the paragraph above.

The authors realize that a drop in ductility at high temperatures has been widely observed. But in all these cases[6] as well as the two given by Mr. Westbrook this embrittlement is not linked to any variable other than temperature. The only suggestion that the embrittlement was strain rate dependent—which was the point raised in this paper—was made by Zay Jeffries to whom we did refer. Jeffries also pointed out that this embrittlement depended on grain size. While he did not point out *how* this embrittlement depended on grain size and strain rate, research in progress in this laboratory indicates that these variables have a strong effect on the temperature at which the embrittlement occurs. The effect is so strong, as a matter of fact, it would be dangerous to state that it occurs at "very near one-half" or any other fraction of the melting point on the absolute temperature scale.

Mr. Westbrook further raises the question of the value for the intercept of the extrapolated strain stabilized branch of the retained ductility - prestrain curve on the retained ductility axis. The data previously presented on steel indicated that this extrapolation yields the same value at zero prestrain in the absence of the rheotropic impediment as was obtained from an extension of the super transition branch of the ductility-testing temperature curve. This indicates, of course, that the curves obtained by prestraining at a variety of super-transition temperatures and testing at a constant low temperature would produce curves of the type shown schematically in Fig. 12. These extrapolated ductility values are thought to be proportional to the prestrain ductility, as shown by the closed dots in Fig. 9, only when the prestraining is at a temperature within the transition range. As is pointed out in the text, under this condition of prestrain full advantage cannot be taken of the rheotropic recovery.

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[6] A list of references pertaining to the subject is appended below.

REFERENCES

D. H. Ingall, "The High Temperature-Tensile Curve: (a) Effect of Rate of Heating: (b) Tensile Curves of Some Brasses," *Journal*, Inst. Metals, Vol. 34, pp. 171–182 (1925).

W. Broniewski, and K. Wesolowski, "L'Influence de la Temperature sur les Proprietes Mechanique de Laitons," *Revve Metallurgie*, Vol. 30, pp. 396–401, 453–457 (1933).

A. Krupkowski, "Proprietes Mechanique du Cuivre, "*Revve Metallurgie*, Vol. 28, pp. 598–609, 641–652 (1931); Vol. 29, pp. 74–92 (1932).

T. Martin, "The Tensile Properties of Aluminum at High Temperatures," *Journal*, Inst. Metals, Vol. 31, pp. 121–152 (1924).

D. H. Ingall, "The Relationship Between Tensile Strength, Temperature, and Cold Work in Some Pure Metals and Single Solid Solutions," *Journal*, Inst. Metals, Vol. 32, pp. 41–64 (1924).

P. Ludwik, "Festigkeitseigenschaften und Molekularhomologie der Metalle bei hohern Temperaturen," *Zeitschrift des Vereines deutscher Ingenieure*, Vol. 59, p. 657 (1915).

R. Stribeck, "Der Warmzerreissversuch von langer Dauer," *Zeitschrift des Vereines deutscher Ingenieure*, Vol. 47, p. 559 (1903).

G. Hughes, "Non-ferrous Metals in Railway Work," *Journal*, Inst. Metals, Vol. 6, p. 74 (1911).

K. A. Huntington, "The Effect of Temperatures Higher than Atmospheric on Tensile Tests of Copper and its Alloys, and A Comparison with Wrought Iron and Steel," *Journal*, Inst. Metals, Vol. 8, p. 126 (1912).

T. Inokuty, "Tensile Tests of Steels at High Temperatures," *Scientific Reports*, Tohoku Imp. Univ., 17, pp. 791–817 (1928).

Z. Jeffries, "Effect of Temperature, Deformation and Grain Size on the Mechanical Properties of Metals," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 60, pp. 474–562 (1919).

M. Rudeloff, "Uber der Einfluss der Warme, chemischen Zausammensetzung und mechanischer Bearbeitung auf die Festigkeitseigenschaften von Kupfer," *Mitteilungen ades dem konigbcher technischr Versuchsanstalten*, Berlin, (1898).

F. M. Howell and D. A. Paul, "The Properties of Aluminum and Two of its Alloys at Elevated Temperatures," *Metals and Alloys*, Vol. 5, pp. 176–179 (1934).

C. H. Lorig, F. M. Dahle, D. A. Robers, "The Mechanical Properties of Copper at Elevated Temperatures," *Metals and Alloys*, Vol. 9, pp. 63–72 (1938).

F. M. Howell and D. A. Paul, "Properties of Wrought Aluminum Alloys at Elevated Temperatures," *Metals and Alloys*, Vol. 6, p. 284 (1935).

W. Broniewski and J. Szreniawski, "L'Influence de la Temperature et de l'Escrouissage sur les Properties Mechaniques d'un Bronze pour Medailles," *Revue de Metallurgie*, Vol. 33, p. 442 (1936).

W. B. Price, "Properties of Copper and Some of Its Important Industrial Alloys at Elevated Temperatures," Symposium on Effects of Temperature on the Properties of Metals, Am. Soc. Testing Mats., p. 340 (Symposium issued as a separate publication *STP No. 12*).

K. E. Quier, "Note on Short-Time High-Temperature Tensile Tests of Wrought Ni-Cr Alloys," *Metals and Alloys*, Vol. 9, p. 40 (1938).

Anonymous, "Mechanical Properties of Copper Cold and Hot, *Cuivre et Laiton*, Vol. 11, pp. 271–274 (1938).

N. K. Czako, "The Mechanical Properties of Metals at High Temperatures," *Metallborse*, Vol. 15, pp. 1352–1353, 1408–1409 (1925).

THE SCALING OF ZIRCONIUM IN AIR*

By C. A. Phalnikar and W. M. Baldwin, Jr.[1]

Synopsis

Both oxygen and nitrogen enter into the scaling reaction of zirconium in air. Given enough time zirconium forms a double-layered scale: an outer white or buff scale (monoclinic ZrO_2) that predominates at temperatures below 1050 C and an inner black scale (monoclinic and tetragonal ZrO_2, cubic ZrN, and possibly nitrogen) that occupies the greater thickness at temperatures above 1050 C. An unusual feature is that the outer white layer does not form immediately but requires a definite time to nucleate. This time is as high as 100 hours near 400 C and less than 5 minutes at 1300 C.

A second bizarre feature is reported: zirconium strip undergoes extraordinary dimensional changes during scaling. Increases in dimensions in the rolling plane trebled the original sample area in some cases. This phenomenon did not set in immediately on heating, but also required a definite time to commence. These times were, in general, much greater than those required to nucleate the outer white scale layer, except in the temperature range 850 C to 1050 C where the two times coincided.

A Pilling and Bedworth parabolic relationship between weight gain and time is roughly followed before the advent of the white outer scale. The appearance of the white scale layer increases the scaling rate enormously at low temperatures, but not at high temperatures.

This paper reports on the scaling behavior of zirconium in air.

Since nitrogen plays as important a part as oxygen in the attack of air on zirconium, the equilibrium diagrams for both of these gases and zirconium, so far as they are known, are shown in Figs. 1(*a*) and 1(*b*).

There are three special points of interest in the scaling of zirconium: the nature of the scale (which changes both with temperature and time in a complicated fashion), the dimensional changes which the metal suffers (and these can be so considerable as to triple the specimen's area), and the reaction rates (which not only reflect the complexities of a chameleon scale and a dilating metal, but evidence some peculiarities *per se*). Fragmentary observations on these odd behaviors have been reported, and Table I assembles the data as coherently as possible. None of these peculiarities, however, has been systematically studied, nor has the correlation between them been properly established. The present paper attempts to correct this situation.

Materials and Procedure

The zirconium sheet was produced by the U. S. Bureau of Mines by reduction

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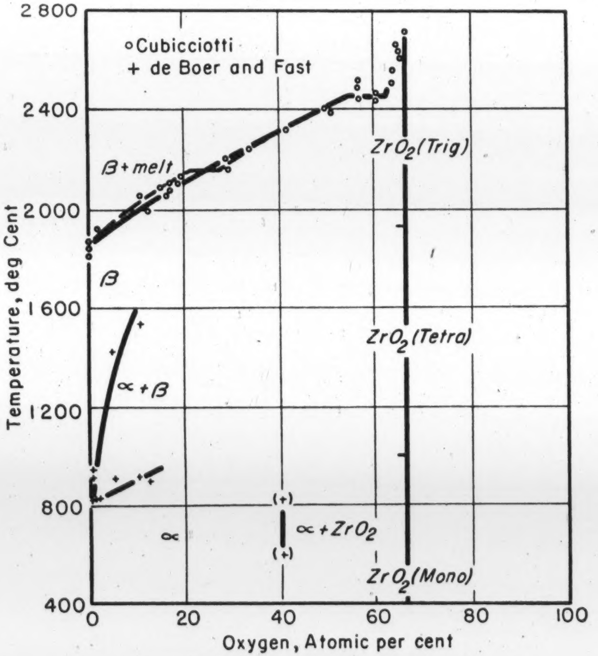
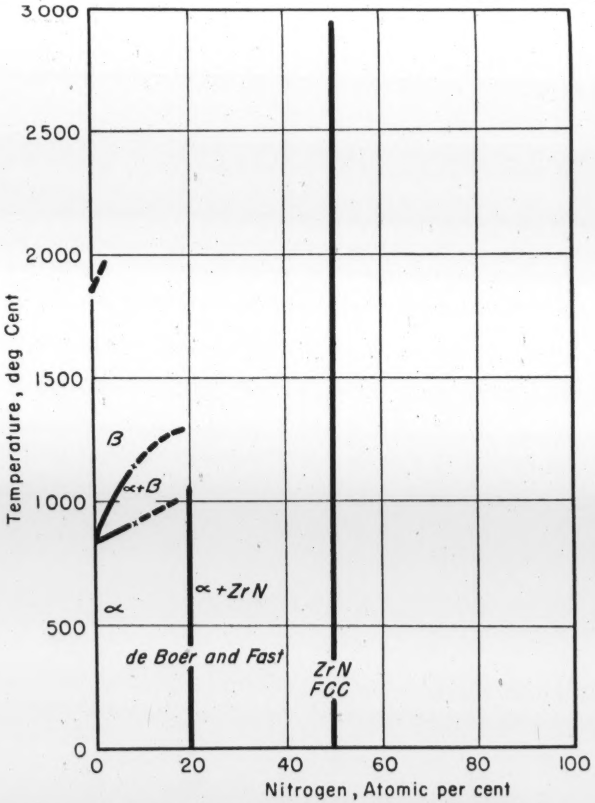
* Presented at the Fifty-fourth Annual Meeting of the Society, June 18–22, 1951.

[1] Graduate student and Research Professor respectively, Department of Metallurgical Engineering, Case Institute of Technology, Cleveland, Ohio.



(*a*) The Zirconium-Oxygen System.

Alpha zirconium is hexagonal close-packed (a = 3.225 Å, c = 5.123 Å); beta is body centered cubic (a = 3.61 Å). Alpha zirconium takes as much as 40 atomic per cent oxygen in terminal solid solution according to de Boer and Fast (**1**) though these authors do not state at what temperature. The $\alpha \rightleftarrows \beta$ transformation curves indicated in the figure actually represent the maxima and minima on electrical resistivity *versus* temperature curves (**1**). De Boer and Fast suggest that the actual transformation curves should fall lower than the lower curve and higher than the upper curves. Cubicciotti (**2**) reports data for the solidus curve, which he suggests, indicate an immiscibility gap about 60 atomic per cent oxygen and that near the melting points only two solid phases are stable: zirconium and zirconium dioxide. His data could allow other phases near the melting point as the dashed lines indicate. The various forms of zirconium dioxide are: monoclinic (a_0 = 5.21 c_0 = 5.375) β = 80 deg. 32 min. tetragonal (a_0 = 5.07 c_0 = 5.16) (this form can be retained at room temperature on cooling), and trigonal (a = 3.598 c/a = 1.633). A cubic a_0 = 5.103 and hexagonal crystal structure have also been reported.



(*b*) Zirconium-Nitrogen Constitution Diagram.

De Boer and Fast (**1**) report the limit of solubility of N_2 in α Zirconium to lie at 20 atomic per cent N_2, and exceeding this limit, face-centered cubic, zirconium nitride (ZrN) a_0 = 4.56 Å is formed. ZrN is stable up to 2930 C.

FIG. 1.—Equilibrium Diagrams for Oxygen, Nitrogen, and Zirconium.

TABLE I.—REVIEW OF FACTS REPORTED ON THE SCALING BEHAVIOR OF ZIRCONIUM IN VARIOUS GASES.

	Hayes and Roberson (3) U. S. Bureau of Mines, Magnesium reduction of zirconium chloride	Gulbransen and Andrew (4) Foote Mineral Co., Thermal dissociation of zirconium tetraiodide	Cubicciotti (5) Foote Mineral Co., Thermal dissociation of zirconium tetraiodide	Dravnieks (6) Foote Mineral Co., Thermal dissociation of zirconium tetraiodide
Nature of Scale:				
In Oxygen	Scales formed from 500 to 900 C were blue-black at first but later showed traces of white oxide. At 1000 C zirconium burns in oxygen.	Scales formed from 300 to 600 C X-rayed at room temperature were monoclinic ZrO_2.	Scales formed from 600 to 900 C were shiny black at first, later white. X-rayed at room temperature scale appeared to be a mixture of both monoclinic and tetragonal ZrO_2 irrespective of color.	
In Nitrogen	Scales formed below 800 C "so thin that surface finish of the cold-rolled sheet is still evident." Above 800 C scale thickens and is golden color. N.B. Scale takes on blue-black discolorations when oxygen is present.	No external scale up to 800 C and 150 min. Nitrogen dissolved directly into metal.		External scales formed from 862 to 1043 C which X-rayed at room temperature, appeared to be ZrN.
In Air	Scales formed were blue-black at first, turned white later. Sample completely white in 24 hr at 700 C, 4 hr at 800 C or few minutes at at 900 C.			
Dimensional Changes:				
In Oxygen	None	None	None	
In Nitrogen	None	None		"Measurable" changes in dimensions after "prolonged" heating at unspecified temperature.
In Air	In 1 hr at 900 C in moist air (20 to 30 per cent humidity) Sample showed 15 to 18 per cent "increase in dimensions." In 1 hr at 900 C in dry air sample showed 8 to 10 per cent increase.			
Reaction Rates:				
In Oxygen	Weight increases given for single times for temperatures from 500 to 900 C.	Weight increase, W, roughly follows Pilling and Bedworth law, between 200 C and 400 C up to 2 hr, ie $W^2 = KT$ where K = constant and T = time. Scaling rates slightly affected by oxygen pressure.	Weight increase, W, roughly follows Pilling and Bedworth law from 600 to 920 C except at 827 and 845 where linear relationship exists. Scaling rates strongly affected by oxygen pressure but in an incoherent fashion.	
In Nitrogen	Weight increases given for single times for temperatures from 700 to 1200 C.	Weight increase, W, roughly follows Pilling and Bedworth law between 400 C and 800 C. Scaling rates were "sensitive to traces of oxygen."		
In air	Weight increases follow Pilling and Bedworth law between 500 and 800 C.			

of zirconium chloride with magnesium. The chemical analysis was not available, but typical analyses for this material are: Fe, 0.05 per cent; C, 0.12 per cent; O_2, 0.10 per cent; and Hafnium; 2 per cent. The ingot was hot-rolled (with a protective iron sheath) at 850 C to 0.1 in. thickness and then cold-rolled to 0.063 in. Specimens approximately 1 by 1 in. were sheared from this sheet. The specimens were washed with dilute sulfuric acid. Weight increase of the specimens as a function of time was determined either continuously by hanging one specimen in a furnace from one arm of a balance, or at intermittent times by putting a number of weighed specimens in a furnace and removing them one by

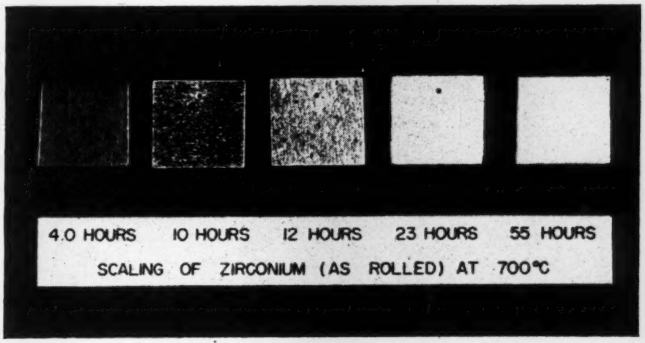


FIG. 2.—Zirconium Scaled in Air at 700 C.

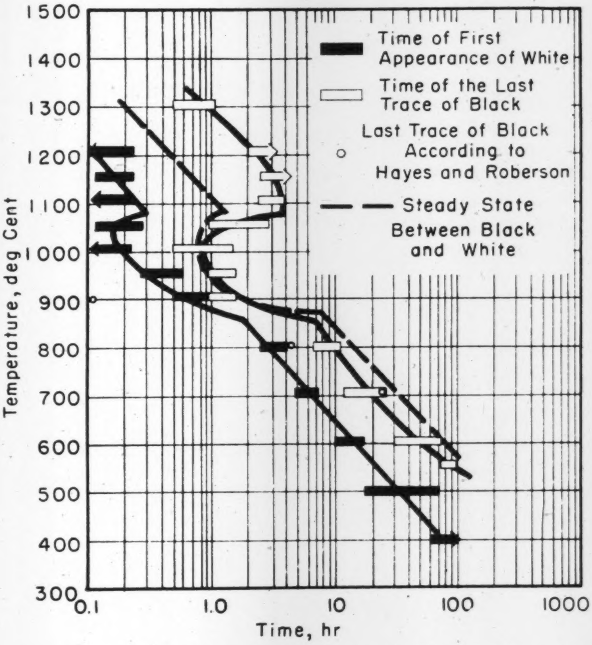


FIG. 3.—Temperature-Time Relationships for the Change in External Appearance of Scale Formed on Zirconium Heated in Air.

one at different times for weighing. A temperature range of 400 C to 1300 C was studied. Weight gain measurements erred by ±0.0001 g and temperature measurements by ±5.0 F.

The scales and the metal were studied micrographically and with X-rays at room temperature.

RESULTS

Nature of Scale:

During the course of isothermal heating in air, the external appearance of the scale changes. The specimens scaled at shorter times have a black metallic scale which becomes covered at longer times with a light layer. At the lower temperatures (up to about 800 C) the layer is buff and first appears both as a bloom or mist and scattering of finely divided flecks. Gradually, the buff layer completely covers the surface. This is easily seen in Fig. 2, a photograph of a series of specimens scaled at 700 C.

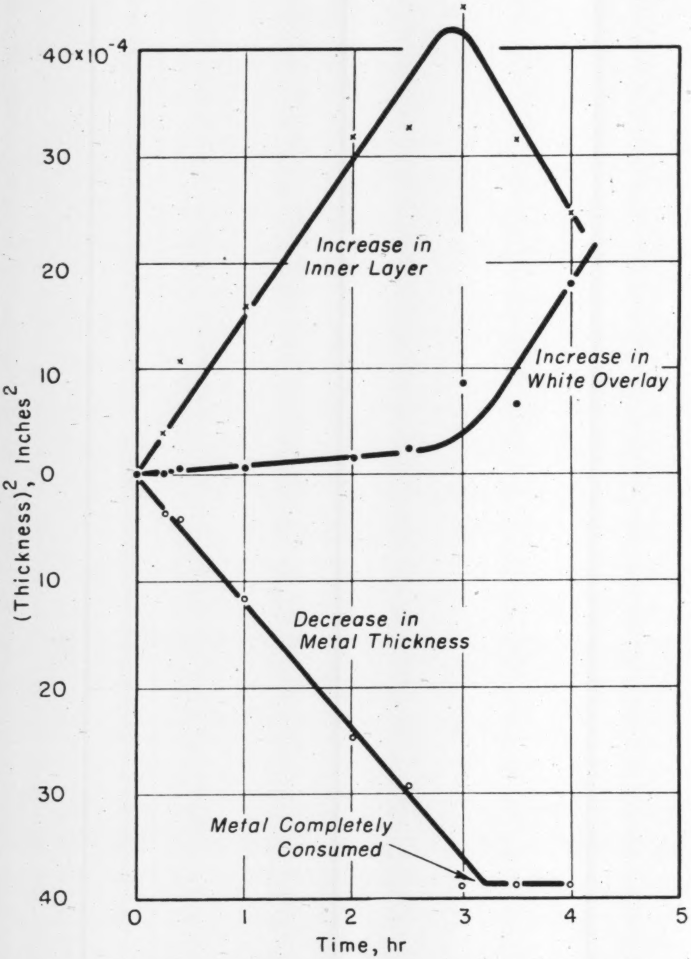


FIG. 4.—Relationship Between Scale and Metal Thicknesses and Scaling Time for 0.063 in. Thick Zirconium Heated in Air at 1100 C.

As temperature is increased (800 C and up) the light scale—now pure white instead of buff—first appears over the black scale as a venous network which gradually fills in until the surface and edge are completely white.

The time at which the light-colored outer layer first appears and finally covers the entire surface shortens as the scaling temperature is increased. Figure 3 is a graph showing these critical times (on a logarithmic scale) as a function of temperature. The lines in this chart show a bulge towards shorter times in the temperature range of 850 C to 1050 C.

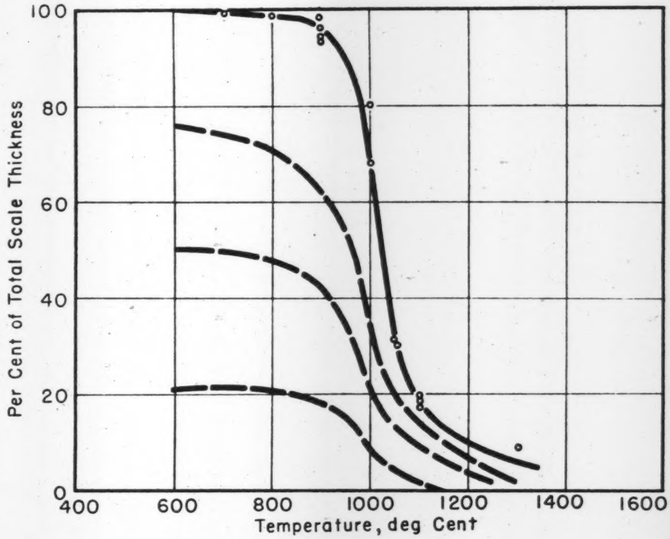


FIG. 5.—Thickness of White Scale as a Function of Temperature.

The data points and solid line represent a steady state. The dashed lines indicate qualitatively how the white scale appears in smaller proportion at times prior to assumption of steady state, thus at 800 C, the scale is all black at first but after some hours acquires an overlay of light scale that grows to increasing proportions of the total scale thickness until at 14 hrs. (see Fig. 3) it occupies 98 per cent of the total thickness.

Thickness measurements of both inner and outer scale layers were made on the microscope. Figure 4 is a plot of the square of thickness of the two-scale layers as well as the metal itself as a function of time for specimens heated at 1100 C. It is seen in this particular case that soon after the white layer makes its appearance the ratio between the thicknesses of the two layers is fairly constant (a steady state). Once the metal is completely consumed, the thickness of the white layer grows at an increased rate, but this of course no longer represents a steady state between two competitive scaling mechanisms. The relative thicknesses of the dark inner layer and white outer layer once a steady state has been achieved is plotted as a function of temperature in Fig. 5. It is obvious that the white scale forms the major portion of the total scale at temperatures below 1050 C but that it represents a minor part above this temperature. At those temperatures where the white scale is predominant, a steady state is not achieved until some time after the exterior of the scale is completely white. At those temperatures where the white scale is a minor part of the total scale, a steady state is reached before the exterior is dead white. The thinness and transparency of the white layer in these cases explains this fact. The times when a steady state is reached is shown in Fig. 3 as a dashed line.

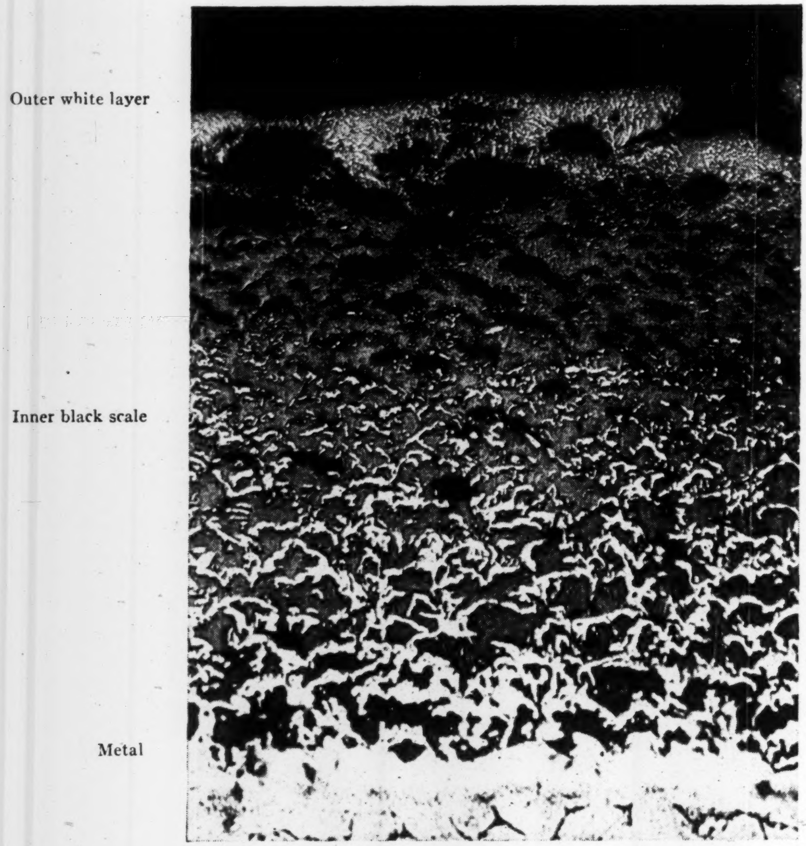


FIG. 6.—Section Through Scale Formed on Zirconium when Heated 1 hr in Air at 1100 C (× 300).

Etched in HF, glycerin, and water. Metal is at the bottom of the photograph. The heterogeneous scale above it is the black scale seen with the unaided eye. This scale contains ZrO_2 as a slate-blue matrix, ZrN as a golden or brass colored filigree in the half of the scale toward the metal, and holes in the outer half of the scale.

The fact that the scales formed on zirconium are double-layered and that (once a steady state may be assumed) the relative thickness of the two layers is a function of temperature is not unique. Iron (**7**)[2], copper (**8**). manganese (**9**), and cobalt (**10**) are known to behave in this manner. Valensi (**8**) has rationalized this behavior, and his theory of scaling mechanism for such cases applied to copper is in good accord with the facts though the case of other metals is not adequately explained. The novel feature of zirconium lies in the fact that the outer white or buff layer does not commence growing simultaneously with the inner layer.

The appearance of the double-layered scale under the microscope is shown in Fig. 6, a photomicrograph at 300-fold magnification of a section through the scale formed in 1 hr at 1100 C. X-ray analysis indicates the outer white layer of Fig. 6 to be monoclinic ZrO_2. The inner layer on the other hand is heterogeneous, being composed of a slate-blue matrix (a mixture of tetragonal and monoclinic ZrO_2, by X-ray analysis) in which there is a regular distribution of holes in the outer half and a golden phase (ZrN by X-rays) in the inner half. Although to the unaided eye the demarcation between the outer white scale and the inner black scale is sharp enough, the boundary between the two scales when viewed at high magnification (such as in Fig. 6) appears diffuse—a fact that is undoubtedly due to the close similarity in crystal structure between the white scale and the slate-blue matrix of the inner scale. The fact that the holes in the outer half of the inner scale have a shape and distribution similar to that of the zirconium nitride particles found at the metal side of the inner scale suggests that they are fossils formed by the decomposition of the nitride. This interpretation is supported by the observation of specimens scaled at 1300 C until the metal was completely consumed: the holes replaced the nitride

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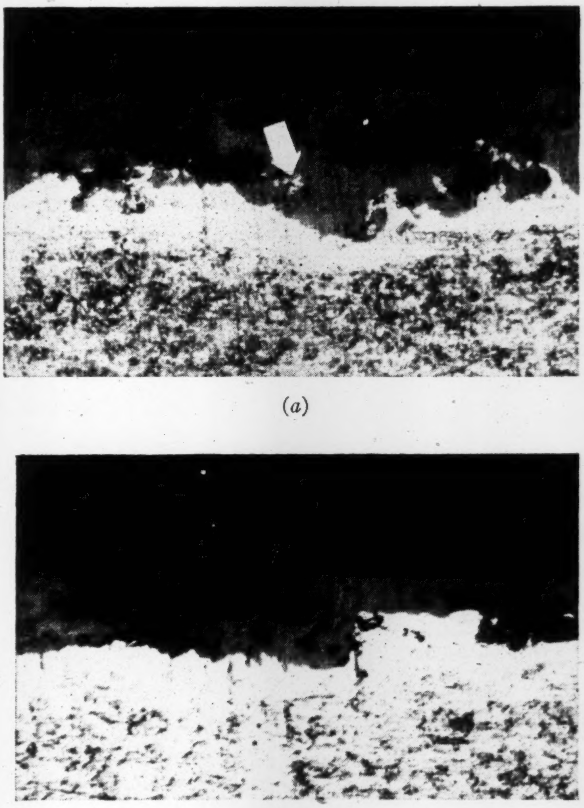
[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1058.


(*a*)

(*b*)

Fig. 7.—Photomicrographs Through Black Scale Formed on Zirconium when Heated in Air to (*a*) 800 C and (*b*) 700 C.

Zirconium nitride particles can still be seen at 800 C as indicated by the arrow. These samples have not been scaled long enough to produce the white outer layer.

phase completely. The fact that the holes occur in all instances right out to the outer edge of the inner layer implies that the scaling does not occur by the outward diffusion of zirconium exclusively (since in this event the newest scale is the outermost region of the layer) but that there is some inward diffusion of the cations. At lower temperatures the appearance of both the scale layers is somewhat different from their appearance at high temperatures. The outer layer becomes tinted: to the unaided eye the scale is buff-colored but under the microscope at high magnification it became rose or mauve. The inner layer, much thinner now, shows less and less of the nitride and similarly fewer holes. The nitride is always confined toward the metal side of the layer. In scales formed at 800 C (Fig. 7(*a*)) only an occasional fleck of the nitride was noticed, and none was found in scales formed at 700 C or lower (Fig. 7(*b*)). It will be recalled from Table I that the external nitride coat disappears when zirconium is heated in pure nitrogen below this same temperature range.

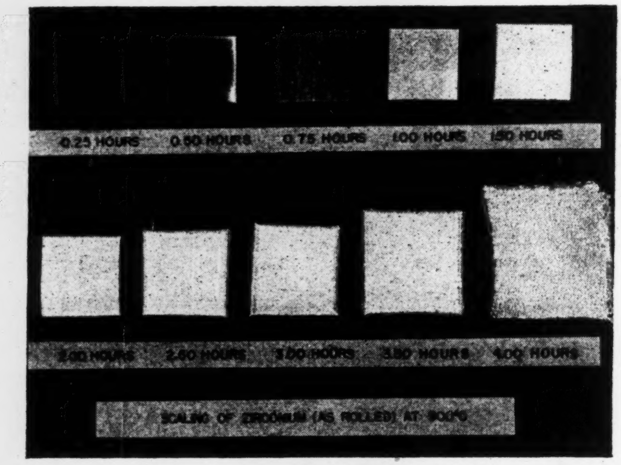


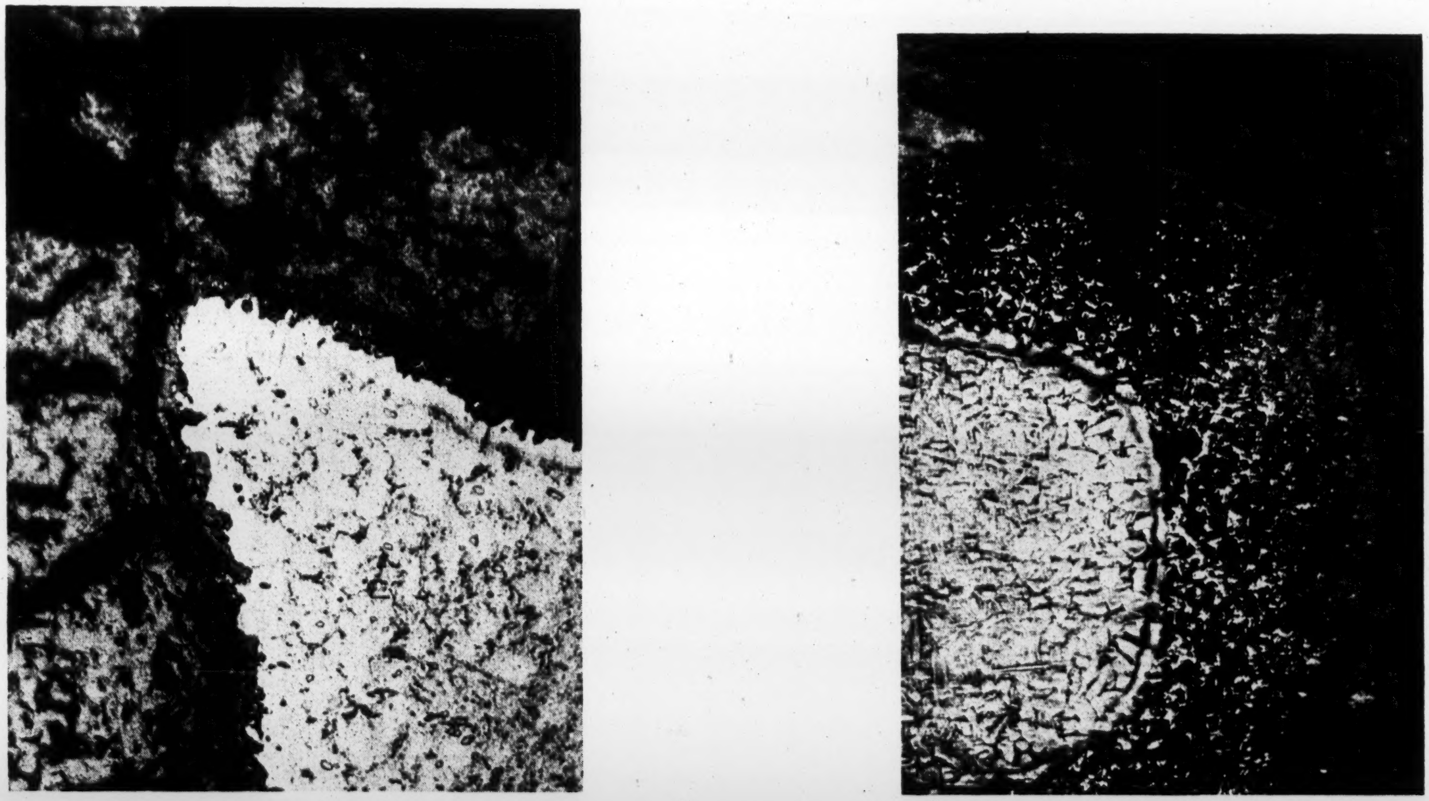
FIG. 8.—Zirconium Scaled in Air at 900 C, Showing Increase in Specimen Area. All samples were of same original size (1 by 1 in.)

Dimensional Changes of the Metal:

In describing the change in the outer white scale as a function of time and temperature, it was mentioned that the lines showed a bulge toward shorter times in the temperature range of 850 to 1050 C (Fig. 3). This bulge is due to the accelerating influence of still another unusual feature found in the scaling of zirconium; the dimensional changes that the specimen evidences.

Figure 8 is a photograph of a series of specimens scaled at 900 C. These specimens were scaled while lying on their side on a ceramic support having a slight dish. The lateral dimensions of the specimen have obviously increased; in fact, increases in area up to 200 per cent over the original area were observed in some series. The increase in lateral dimensions is definitely an increase of the dimensions of the metal and not the scale alone. Figure 9 is a photomicrograph of a section through the edge of the specimen: the metal is seen to come right out to the edge.

Dravnieks (6) describes this as a "swelling" of the zirconium metal and


White scale
Black scale
Metal

(*a*) Edge that showed 40 per cent increase in area when scaled 2 hr at 900 C.

(*b*) Edge that did not show lateral growth when scaled for 2 hr at 1100 C.

Fig. 9.—Photomicrographs of Edges of Scaled Metal.

says its is due to the "volume increase caused by nitrogen when the latter approaches saturation in the metal." Actually de Boer and Fast's (1) data show increases in parameter at the saturation compositions of oxygen and nitrogen of the order of 1 per cent, which figure hardly explains the 200 per cent lateral expansion found in some cases. Nor can the 2 per cent volume change of $\alpha = \beta$ transformation explain the large changes found. (The fact that the expansion takes place in time at temperatures as low as 600 C which is far below the $\alpha = \beta$ transformation temperatures reported by de Boer and Fast would require some explanation.) It could not be argued defensibly that oxide intrusions into the metal caused the dimensional instability. The Pilling and Bedworth ratio for zirconium to its dioxide is 1.52 and for zirconium to its nitride is 1.088, so that the maximum lateral growth would be less than about 50 per cent even in the extreme event that all the metal were converted to oxide or nitride by an intrusion mechanism. In none of the specimens was more than 5 per cent of the volume of metal found to consist of intrusions, so that the corresponding maximum lateral growth was of the order of 2 to 3 per cent. As a matter of fact, all these explanations imply that the changes observed are a *volume* expansion. X-ray and pycnometric measurements of lattice parameter and specific gravity of the metal after a 200 per cent increase in

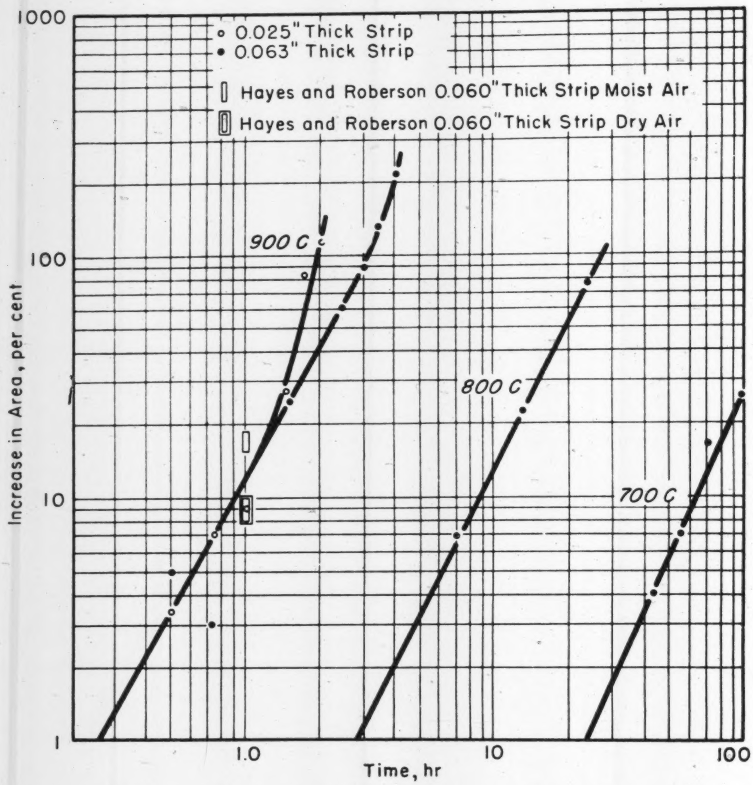


FIG. 10.—Effect of Time on Increase in Area at 700 C, 800 C, and 900 C.

area showed virtually no difference from those on starting material, which indicates that the dimensional changes observed represent a deformation rather than an *expansion*, and in particular a compression in the direction normal to the rolling plane. This raises the question of whether the specimens in their horizontal position flowed by some mechanism such as creep. When specimens were hung from a wire or stood on edge and scaled, they would show some evidence of the stress state that these positions produced, for example, hung specimens would be slightly broader at the bottom edge than the top, but still the top would be broader than the original specimen, which is the opposite effect from that which would be expected for flow under this stress state.[3] Specimens around which nichrome wire had been wrapped would break the wires when scaled; specimens which had two parallel edges clamped would buckle as a result of this restriction and grow in the direction in which they were free. These experiments indicate that the lateral expansion was not a case of simple flow under a stress state induced by external forces.

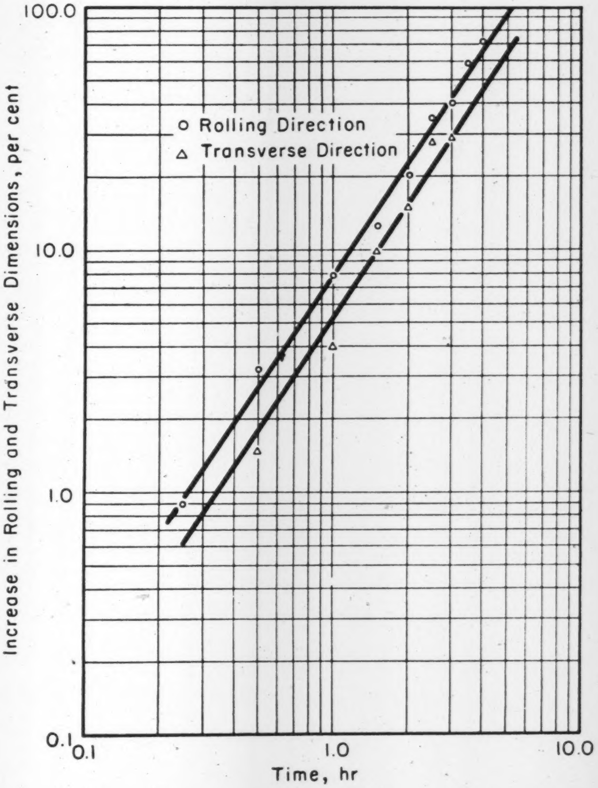


FIG. 11.—Increase in Dimensions of the Rolling and Transverse Directions for Zirconium Heated at 900 C as a Function of Time.

Specimens scaled in pure oxygen or nitrogen at 900 C showed no lateral growth within 4 hr at 900 C (another proof incidentally that the expansion was not a case of simple flow under a stress state induced by external forces), indicating that the growth of the specimens when scaled in air is due to some cumulative action of nitrogen and oxygen.

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[3] Even for anisotropic metal, the reaction of the metal to this stress state by ordinary flow would not lead to a broadening of the top.

The question arises whether there might be some thickness effect, that is, would a zirconium specimen dissolve the critical quantity of nitrogen and oxygen necessary for deformation in less time than would another specimen that was thicker? Test specimens cold rolled from 0.063 in. to 0.025 in. and scaled in air at 900 C gave virtually the same dimensional changes in the same time as did specimens from the original 0.063 in. stock (Fig. 10). If a diffusion-controlled mechanism were operative in the metal, comparable growth should have occurred for the thinner specimens at roughly one-sixth the time for thicker ones.[4]

When specimens were scaled lying on their side it was found that the growth in the rolling direction was always greater (by close to 4.5 per cent) than the growth in the transverse direction. Figure 11 is a graph showing this behavior of zirconium scaled at 900 C.

The time at which growth had reached a 1, 10, and 100 per cent increase in area is plotted on a logarithmic scale as a function of temperature in Fig. 12. It is seen that just as in the case of the color changes, the time required for a given growth decreased as scaling tem-

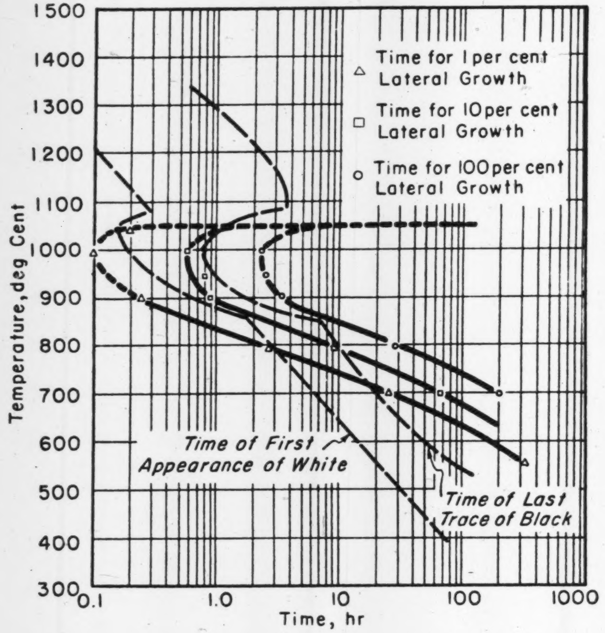

FIG. 12.—Temperature Relationships Involved in Lateral Growth of Zirconium Scaled in Air.

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[4] Thus, if a critical concentration of nitrogen at an arbitrary proportion, p, of the thickness of the metal (X_s for the thin metal, X_L for the thick metal) is necessary for a given expansion, and if this concentration is built up by diffusion, the times required (t_s for the thin sample, t_L for the thick sample) would be

$$\frac{(pXs)^2}{(pX_L)^2} = \frac{t_s}{t_L} = \frac{0.025^2}{0.063^2} \text{ or } t_s = 0.16\, t_L$$

perature increased. The exact relationship does not coincide over all temperature, however. At low temperatures the color change occurs first and then the growth of the metal sets in. At temperatures from 850 to 1050 C the two curves run roughly parallel, and it is felt that the growth of the specimen provokes the outer scale to appear abnormally early. The exact mechanism

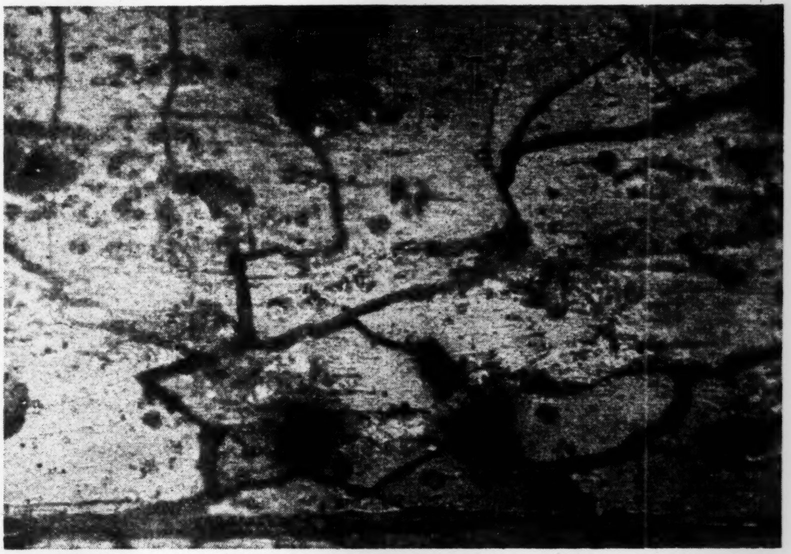


Fig. 13.—Surface of Zirconium Scale Showing Early Cracks (× 500).

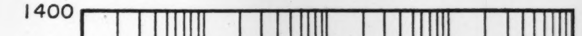
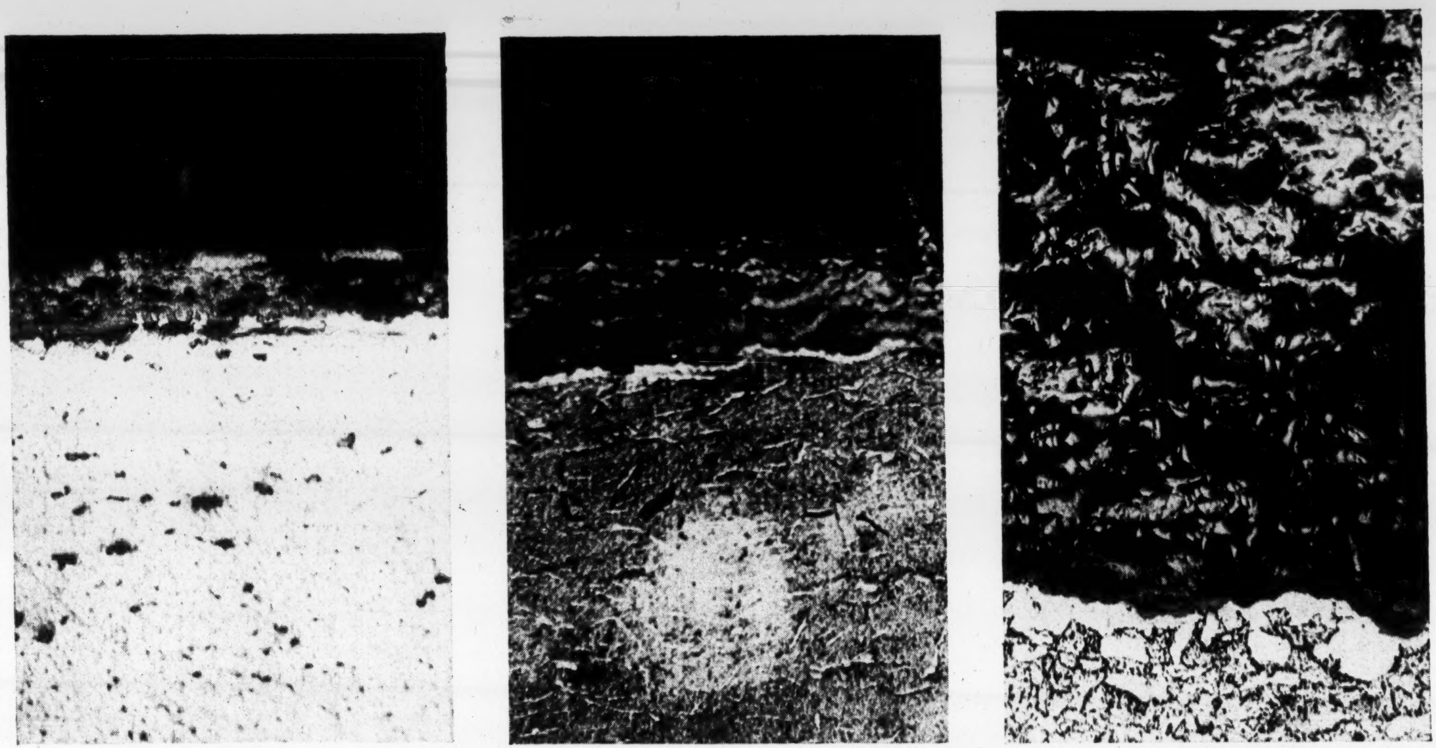


Fig. 14.—Cracking of Scale Surface as Observed at 100-Fold Magnification as a Function of Time and Temperature.



(a) (b) (c)

FIG. 15.—Zirconium Scaled at 800 C for Various Times.

The microstructure in (a) is typical of those found before any observable dimensional changes, in (b) the microstructure is typical of metal which has shown an increase in area of about 5 to 40 per cent, and in (c) the structure corresponds to metal having an increase in area of more than 70 or 80 per cent. The same structures were observed in zirconium scaled at 700 C.

by which this triggering occurs is not evident at the moment: The appearance of the venous network of the white scale on the black surface in this temperature range as opposed to the general bloom in which the white first appears at low temperatures indicates that the mechanism is associated in a general way with the network of cracks which the lateral dilation of the underlying metal produces in the brittle scale, but whether the triggering is due to the creation of sharp points of unstable scale or some other specific mechanism is not clear.

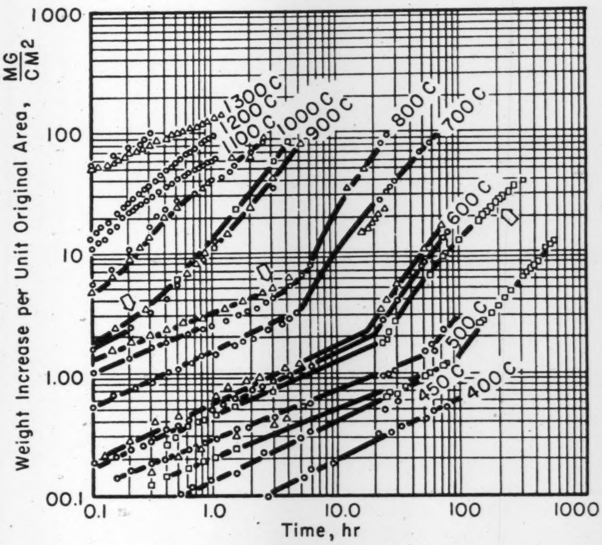


FIG. 16.—Isothermal Weight Increase *versus* Time Curves for Zirconium Heated in Air.

Arrows indicate points where 1 per cent growth occurred.

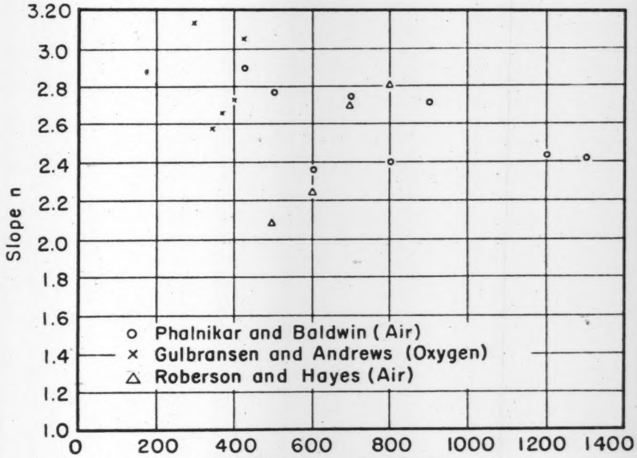


FIG. 17.—Variation of n in the Equation $w^n = Kt$ with Temperature for the Scaling of Zirconium in Air.

The cracking of the scale is obvious in specimens that have grown 100 per cent from their original lateral dimensions, as Fig. 9 shows, but to the unaided eye it is difficult to assess when cracking really begins. Inspection of the surfaces of scales at 500-fold magnification shows cracking of the surface (Fig. 13) occurs when the specimens have grown only a few per cent laterally (Fig. 14).

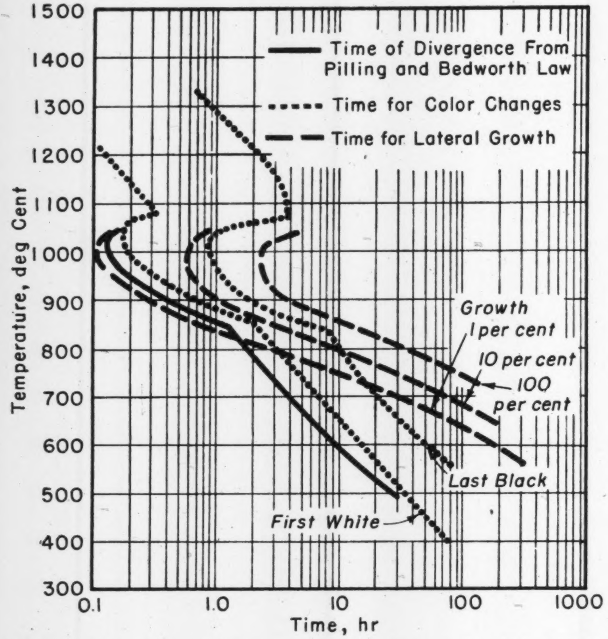


FIG. 18.—Time for Divergence from Pilling and Bedworth Law, for Color Changes and for Lateral Growth as a Function of Temperature.

Specimens which have grown laterally show a distinctive configuration at the edges. The white scale peels away from the edge while the metal (still covered with a thin layer of black-and-gold scale) protrudes with an acute angle contour (Fig. 9(*a*)). Specimens scaled at higher temperatures, however, which showed no growth, had a normal contour at the metal edge (see Fig. 9(*b*)). The microstructure of the metal undergoes a characteristic change during the deformation process. Figure 15 gives three stages of the change observed at 800 C. X-ray analysis using normal diffraction techniques of these three structures as with all other specimens irrespective of scaling temperature or quenching revealed only alpha zirconium.

Weight Measurements:

Weight increase measurements as a function of time reflect these complicated mechanisms (Fig. 16). At low temperatures and short times when the black scale is the only layer formed, oxidation occurs at a slow rate and follows a course that approximates the Pilling and Bedworth parabolic law,

$$w^2 = Kt$$

where:

w = weight increase per unit area,
K = constant, and
t = time.

The weight increase actually rises at a slower rate than called for by the

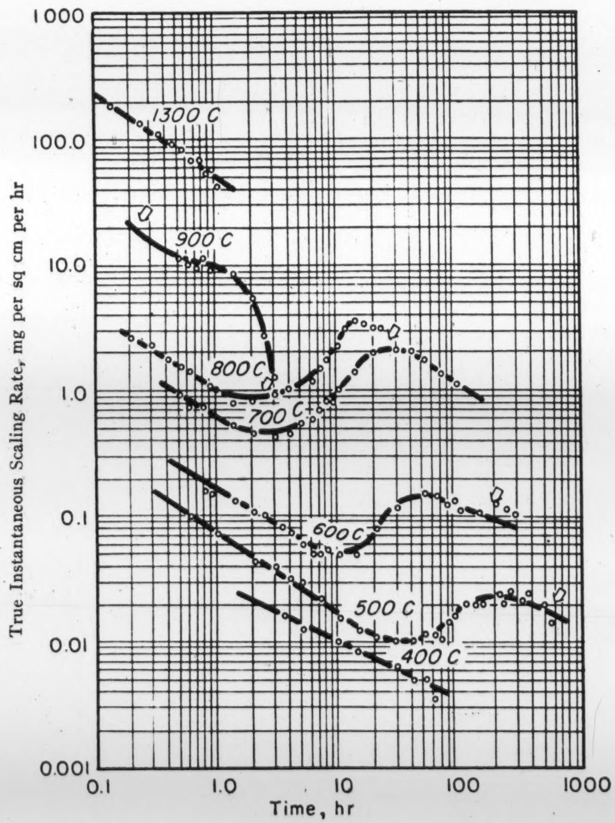


FIG. 19.—True Instantaneous Scaling Rate as a Function of Time and Temperature.

Arrows indicate 1 per cent lateral growth.

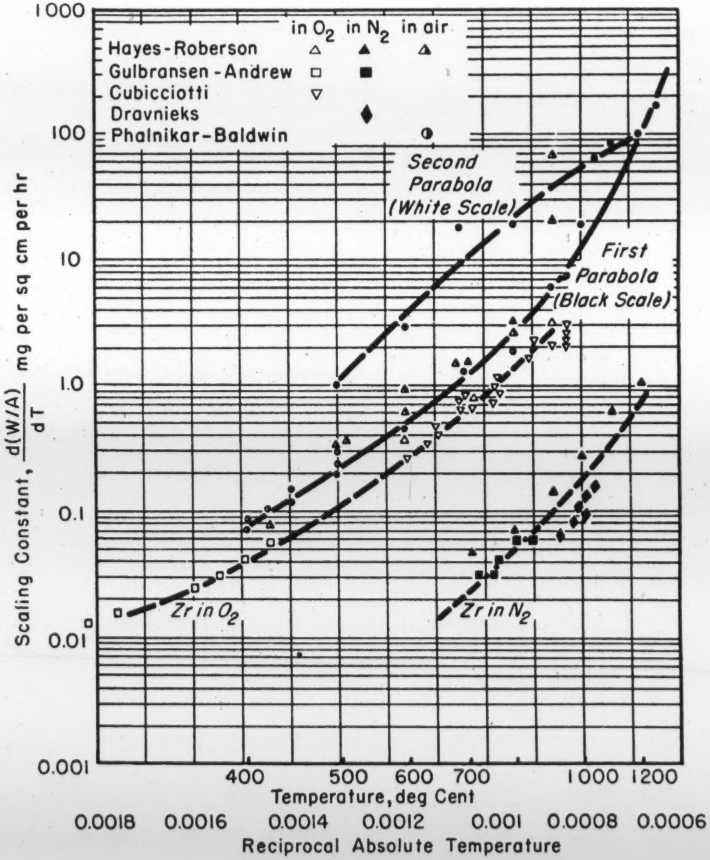


FIG. 20—Arrhenius Plot of Reaction Rate for Zirconium Scaled in Air, Oxygen, and Nitrogen.

Pilling and Bedworth law, which trend is obvious in Gulbransen and Andrews' data also. The data can be expressed by a parabola of more generalized form:

$$w^n = Kt$$

where n is a number other than 2 (see Fig. 17). At temperatures up to 1050 C the weight increase *versus* time data suddenly—at a critical time which depends upon the temperature—depart from the quasi-Pilling and Bedworth curve. The critical times at which this departure occurs is plotted on a logarithmic scale as a function of temperature (Fig. 18). These times coincide with the times at which the outer white scale makes its appearance and not the times at which lateral growth or cracking occurs (except of course in the temperature range of 850 to 1050 C where the latter process coincides with, if not induces, the former process). At temperatures above 1050 C the data follow quasi-Pilling and Bedworth curves without interruption. At these higher temperatures, the white outer layer (see Fig. 5) occupies a small part of the scale and its delayed appearance affects the weight increase curves to a correspondingly small degree.

The fact that the specimens grow affects the analysis of the data presented in Fig. 16. Since the area is changing, the data presented in this figure do not allow a precise determination of the scaling mechanism (that is, the true instantaneous scaling rate). Nor would such a determination be possible, if the weight increase data were recalculated on the basis of unit of instantaneous area, for it is readily seen that weight increase is an integrated function of the two time-dependent functions, (a) the rate of growth of area, $\frac{dA}{dt}$, and (b) the rate of scaling per unit of instantaneous area $\frac{d(W/A)}{dt}$. The true instantaneous scaling rate, F, is obtainable by differentiating W/A with respect to time. That is

$$F = \frac{d\,W/A}{dt} = \frac{A\frac{dW}{dt} - W\frac{dA}{dt}}{A^2} \ldots\ldots (2)$$

$$\text{or, } F = \frac{\frac{A}{A_0}\frac{d\,(W/A_0)}{dt} - \frac{W\,d(A/A_0)}{dt}}{(A/A_0)^2} \ldots\ldots\ldots (2a)$$

where:

W = weight increase, mg,
A_0 = original specimen area, sq cm,
A = final specimen area, sq cm, and
t = scaling time, hr.

Figure 19 is a plot of the true instantaneous scaling rate, F, as a function of time and temperature on log-log scale. A straight line with a slope of -0.5 on this plot will indicate a parabolic law, if no initial scale is present on the metal. If an initial scale is present, the slope will be between 0 and -0.5. A straight line with a zero slope will indicate a linear law, whether the metal possesses an initial scale or not.[5]

This chart, Fig. 16, indicates that at 400 C an approximate parabolic law holds over the whole time range studied. From 500 to 800 C, two successive approximate parabolic laws are obeyed. The wonder here is that at 700 C and 800 C the second parabola continues to hold even after growth of the specimen

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[5] Differentiating the Pilling and Bedworth parabolic law,

$$(W/A)^2 = K_1t + K_2$$

with respect to time gives

$$\frac{d\,(W/A)}{dt} = \frac{K_1}{2\sqrt{K_1t + K_2}}$$

If K_2 is zero this equation reduces to

$$\frac{d\,(W/A)}{dt} = \frac{1}{2}\sqrt{\frac{K_1}{t}}$$

which will give a slope of -0.5 when the derivative and t are plotted on log-log paper. If K_2 is infinite the equation reduces to

$$\frac{d\,(W/A)}{dt} = 0$$

Differentiating the linear law

$$W/A = K_3t + K_4$$

with respect to time gives

$$\frac{d\,(W/A)}{dt} = K_3$$

has set in and cracking of the outer scale occurs (see arrows in Fig. 18). At 900 C the curve is so irregular that no analysis can be made. At temperatures from 1050 C and up a single parabolic law was found.

The values of the reaction rate at 1 hr determined from data plotted as in Fig. 19 have been entered in the Arrhenius chart shown in Fig. 20. It is seen that the data coincide well with Hayes and Roberson's for the scaling of zirconium in air and lie above previous investigators' parabolic scaling constants of zirconium in oxygen by a factor of about 2; whereas, they lie at about two orders of magnitude above the parabolic scaling constants of zirconium in nitrogen. The implication of this and the fact that oxygen and nitrogen are both found in the scale is that the combined presence of the two gases serves to stimulate the scaling rate.

DISCUSSION

There are many unusual points involved in the scaling of zirconium in air that require explanation. Some of these points cannot be explained because of a lack of information on such basic facts as the nature of the ternary zirconium-oxygen-nitrogen constitution diagram, etc. However, other of the points can be given a tentative explanation.

The Delayed Appearance of the White Outer Layer.—This phenomenon cannot be explained at present. It is not unique, however, for a similar behavior has been observed in this laboratory (and will be soon reported) in the case of titanium and lead.

The Temperature Dependence of the Relative Thickness of White Outer Scale and Dark Inner Scale.—This can be explained tentatively. At low temperatures the scaling rate of the zirconium is higher with the white scale; at high temperatures it is lower with the white scale, Fig. 20. As shown by Valensi, the relative thicknesses of the two layers of a double-layered scale will be set by the relative scaling rates of the two individual layers. Of the two layers, that layer with the higher scaling rate will form the relatively thicker scale. It is impossible to apply Valensi's theory quantitatively, however, for the exact amounts of zirconium fixed by the two gasses in the two scale layers is unknown.

The Occurrence of Relatively Large Amounts of Nitride in the Inner Layer.—The rate of scaling of zirconium in pure nitrogen is roughly 1 per cent that of the rate of scaling of zirconium in pure oxygen over the temperature range investigated. How large amounts of nitride are formed (up to 50 per cent at the metal interface according to Fig. 6(*b*)) is unexplained. The possibility that the nitride phase may have decomposed from a solid solution of nitrogen in oxide when cooled from scaling temperatures cannot be ignored. Samples that were quenched from temperature showed exactly the same microstructure as samples that were air-cooled, however, which tends to make this suggestion less likely.

The Occurrence of Holes in the Inner Layer.—Until the ternary diagram Zr-O-N is known, a definite explanation for the holes is lacking. It is known, however, that ZrO_2 is stable toward nitrogen (rather than ZrN being stable toward oxygen); hence the ternary diagram must contain a tie-line between the nitrogen corner and ZrO_2. A line in an isothermal ternary diagram drawn between the zirconium corner with the point (80 per cent N_2, 20 per cent O_2—air) to indicate the various possible phases across the scale layer must cross this tie-line unless it passes through the single-phased ZrO_2 field. The presence of ZrN at the metal interface makes this latter possibility less probable, whence it may be tentatively assumed that the holes originally contained nitrogen.

The Relatively Higher Scaling Rate of Zirconium in Air as Compared with that of Zirconium in Oxygen or Nitrogen.— This can be explained if it is assumed nitrogen dissolves, even though it be to a minute extent, in ZrO_2. Here again knowledge of the ternary diagram would be desirable, yet the assumption is quite probable in view of the presence of ZrN at the metal interface of the inner scale layer. Since nitrogen is quadrivalent, it would create defects in the oxygen ion structure, thus permitting a higher rate of diffusion of oxygen through the ZrO_2. An analogous suggestion has been put forward by Wagner (**11**) for the increased scaling rate of nickel-chromium and nickel-manganese alloys over that of pure nickel,[6] where the trivalent chromium dissolving in NiO creates defects in the divalent nickel ion structure, thus allowing nickel to diffuse through the oxide at a higher rate. It will be noticed in the present case the explanation of an increased scaling rate on the basis of an increased diffusion rate of *cations* assumes that the scaling mechanism occurs, at least in part, by the inward diffusion of cations. This is, however, supported, as noted above, by the fact that the nitride is at the inner side of the inner scale and the holes are on the outer part of the inner scale.

The Dimensional Changes of the Metal. —The deformation of the metal appears to be related to microstructural changes occurring within the metal. The only causes for the deformation apparent to the authors is a gross coherent change in crystal structure or orientation. In either case, this necessarily implies the presence of a gross coherent orientation in the original metal (which is common in rolled hexagonal metals). The directionality in lateral growth reported in Fig. 12 also supports this deduction. The only means by which the change could take place is by a transformation or by recrystallization. The fact that normal X-ray patterns show nothing but alpha zirconium does not eliminate the possibility of the transformation.

Acknowledgment:

The authors are especially indebted to the Research Corporation whose grant-in-aid made this work possible. They wish to thank O. C. Ralston, Chief, Metallurgical Research, of the Bureau of Mines for the zirconium used in this thesis. Mr. Stephen Mayersky performed the metallographic work for this report.

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[6] Experimentally copper-manganese and copper-chromium alloys are found to scale more rapidly than pure copper and a similar explanation can be put forth here (**12**.)

REFERENCES

(**1**) J. H. de Boer and J. D. Fast, "The Influence of Oxygen and Nitrogen on the α-β Transition of Zirconium" *Recueil des travaux Chimiques des Pays-Bas,* Vol. 55, p. 459 (1936).

(**2**) D. Cubicciotti, "The Melting Point—Composition Diagram of the Zirconium-Oxygen System," ONR Research Contract N7-ONR-329 Task Order II *Technical Report No. 6,* Illinois Institute of Technology (1950).

(**3**) E. T. Hayes and A. H. Roberson, "Some Effects of Heating Zr in Air, Oxygen and Nitrogen," *Transactions,* Electrochemical Soc., Vol. 96, pp. 142–151 (1949).

(**4**) E. A. Gulbransen and K. Andrew, "Kinetics of the Reactions of Zirconium with O_2, N_2 and H_2," *Transactions,* Am. Inst. Mining and Metallurgical Engrs., Vol. 185, pp. 515–525 (1949).

(**5**) D. Cubicciotti, "The Oxidation of Zirconium at High Temperatures," ONR Research Contract N7-ONR-329 Task Order II *Technical Report No. 4,* Illinois Institute of Technology.

(**6**) A. Dravnieks, "The Kinetics of Zirconium-Nitrogen Reaction at High Temperatures," ONR Research Contract N7-ONR-329 Task Order II *Technical Report No. 5,* Illinois Institute of Technology (1949).

(7) J. Benard and O. Coquelle, "Nouvelles recherches par la methode micrographique sur l'oxydation du fer aux temperatures elevees," *Comptes rendue,* Vol. 222, pp. 796-79 (1946).

O. A. Tesche, "An X-Ray Study of the Scale Formed on Iron between 400 and 700 C," *Transactions,* Am. Soc. Metals, Vol. 42, pp. 641-648 (1950).

J. K. Stanley, J. von Hoene, and R. T. Huntoon, "The Oxidation of Pure Iron," *Transactions,* Am. Soc. Metals, (1950).

(8) G. Valensi, "Theoretical and Experimental Investigations about Conjugated Formation of Several Layers of Dry Corrosion," Pittsburgh International Conference on Surface Reactions, Corrosion Publishing Co., Pittsburgh 12, Pa. 1948, pp. 156-164.

(9) R. S. Gurnick and W. M. Baldwin, Jr., "The High Temperature Oxidation of Manganese," *Transactions,* Am. Soc. Metals, Vol. 42, pp. 308-318 (1950).

(10) C. R. Johns and W. M. Baldwin, Jr., "High Temperature Scaling of Cobalt," *Transactions,* Am. Inst. Mining and Metallurgical Engrs., Vol. 185, pp. 720-721 (1949).

(11) C. Wagner and K. E. Zimens "Die Oxidationsgeschwindigkeit von Nickel bei Kleinen Zusatzen von Chrom and Magan," *Acta Chemica Scandinavica,* Vol. 1, pp. 547-565 (1947).

(12) K. W. Frohlich, "Die Zunderung von reinem und von legiertem Kupfer," *Zeitschrift fur Metallkde,* Vol. 28, pp. 368-375 (1936).

DISCUSSION

MR. M. M. JACOBSON.[1]—Have the authors made any observations concerning the relative adhesion or hardness of the zirconium scale layers?

MR. W. M. BALDWIN, JR. (*author*).—Any answer I give you must be tempered by taking into consideration both the growth of the metal and the change in scale. The black scale is extremely adherent to the metal while the white scale is flaky.

When the metal grows it continually cracks the scale so that the adherence is rather poor.

We made no hardness measurements on the scales themselves. Of course, there are studies of these oxides and nitrides in the literature; in general they are very hard.

MR. P. H. MORTON[2] (*by letter*).—As in the case of other properties, the oxidation behavior of zirconium appears to be quite similar to that of titanium. In the case of titanium, the monoxide TiO is formed at temperatures above about 800 C. Is it possible that an analogous ZrO structure may exist at some temperatures, and that the golden phase observed by the authors is not the nitride but the oxide? In the case of titanium the TiO has a similar color and the same NaCl structure as the nitride, TiN. Further there is a considerable range of composition for both oxide and nitride, and the lattice parameter varies with both composition and temperature. This makes it very difficult to distinguish between oxide and nitride if X-rays are used alone. Indeed it seems probable that a considerable, if not complete, range of solid solution exists between TiO and TiN. The golden phase observed in this paper and described as the nitride could alternatively be a monoxide ZrO, or a solid solution of oxygen in ZrN.

The explanation of the holes in the outer half of the black scale as the fossils of decomposed nitride is interesting. If this is the case, then as the authors state, reaction must in part take place by diffusion of the nitrogen inwards. But we can also postulate that the holes form first as nitrogen inclusions and that the zirconium ions diffuse outward and convert them to nitride inclusions. Diffusion of nitrogen ions need not then take place.

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[2] Research Assistant, Metallurgical Engineering Dept., Case Institute of Technology, Cleveland, Ohio.

MR. BALDWIN (*author*).—The points raised by Mr. Morton are excellent criticism. The experimental evidence in the paper can be interpreted as he points out in alternate ways. Until further evidence is developed the questions will have to remain open. The fact that a zirconium monoxide has not been reported certainly does not negate his argument. Indirect evidence supports the interpretation we placed on the two points he has raised: the disappearance of the golden phase at temperatures below 800 C is consonant with the disappearance of an external scale when zirconium is heated in pure nitrogen, which favors the nitride interpretation. The increased scaling rate of zirconium in air over that in pure oxygen favors not only the interpretation of nitrides but also the interpretation of the inward migration of nitrogen as outlined in the paper. If the interpretations which Mr. Morton suggests ultimately turn out to be correct then these pieces of indirect evidence will have to be reinterpreted also.

AN ARBITRATION BAR IZOD IMPACT TEST FOR CAST IRON*

By J. T. Eash[1] and A. P. Gagnebin[2]

Synopsis

An impact test for cast iron, which has been in use for several years, is described, consisting of breaking a section of an unmachined arbitration bar in a modified Izod machine.

Wide interest has been shown in this simple form of impact test, and therefore, to assist others who may be interested in obtaining data in the same numerical system, dimensional constants of the machine are presented. Illustrative data are included which show that the system accomodates various types of cast iron with impact values ranging from 10 ft-lb for high phosphorus gray irons to over 120 ft-lb for austenitic cast irons. The impact results demonstrate the usefulness of the method both to develop the optimum composition and melting technique for new alloys and to assist in the selection of the material most suitable for particular engineering applications.

Several years ago, when increasing demands were beginning to be made on cast iron in engineering applications, it became evident that a dynamic test would not only be useful to the metallurgist for developing new cast iron alloys but would also assist the engineer in the better use of existing compositions. The conventional Izod and Charpy systems were inadequate because the small specimens employed had such a low order of impact resistance that they did not serve to distinguish between many of the ordinary gray cast irons, and furthermore these small samples must be selected from some specific location in a casting. A system was needed that would test a complete casting, including the undisturbed skin, instead of a section representing only a portion of the gradient structures. Since the 1.2-in. arbitration bar filled this requirement and was accepted by the foundry industry as a standard specimen, it was only natural that it should be used.

A simple convenient test was devised utilizing a section of the arbitration bar, which is subjected to impact in a modified Izod machine. The method has been in use for over 15 years and has proved its usefulness in plain cast irons, high-strength gray irons, austenitic irons and different types of white irons. In view of the wide interest shown in this simple way of measuring impact resistance, the dimensional constants of the testing machine are presented as well as data illustrating the application of the method in order to assist others who may be interested in obtaining results in the same numerical system.

Design of the Impact Machine

The equipment used is a regular Olsen Izod machine, but with a shorter pendu-

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* Presented at the Fifty-fourth Annual Meeting of the Society, June 18–22, 1951.

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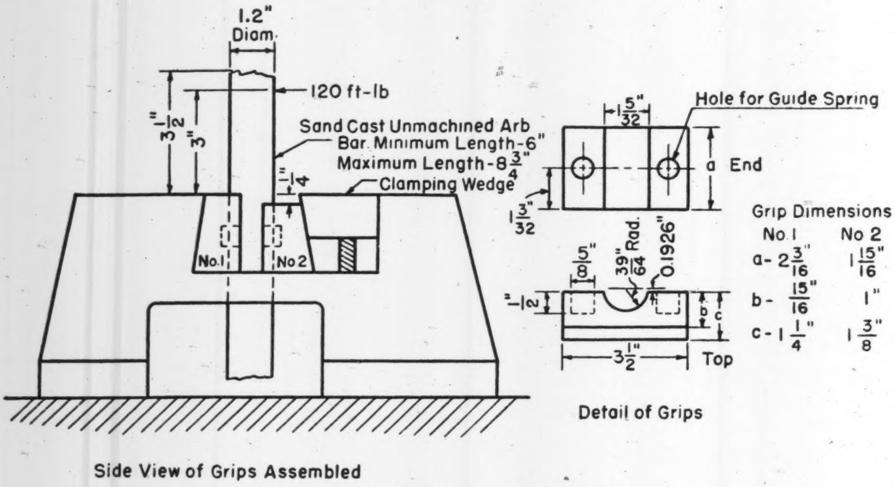
1.2" Diam.
120 ft-lb
Sand Cast Unmachined Arb Bar. Minimum Length-6" Maximum Length-8 3/4"
Clamping Wedge
3 1/2"
3"
1/4"
No.1
No 2
1 5/32"
Hole for Guide Spring
a End
1 3/32
Grip Dimensions
No.1 No 2
a- 2 3/16 1 15/16"
b- 15/16" 1"
c- 1 1/4" 1 3/8"
5/8"
39/64" Rad.
0.1926"
1/2"
b c
3 1/2"
Top
Detail of Grips
Side View of Grips Assembled

Fig. 1.—Grip Assembly and Specimen.

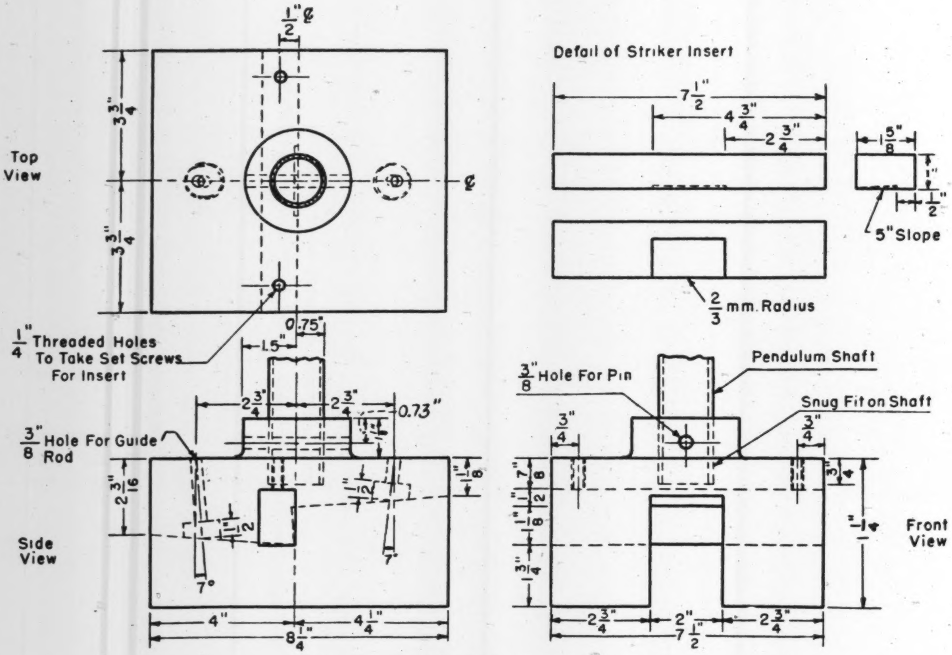
1/2" ℄
Top View
3 3/4"
3 3/4"
℄
1/4" Threaded Holes To Take Set Screws For Insert
0.75"
1.5"
2 3/4"
2 3/4"
0.73"
3/8" Hole For Guide Rod
2 3/16"
1 1/8"
1/2"
1 1/2
7°
7°
Side View
4"
4 1/4"
8 1/4"
Detail of Striker Insert
7 1/2"
4 3/4"
2 3/4"
1 5/8"
1"
1/2"
5" Slope
2/3 mm. Radius
Pendulum Shaft
3/8" Hole For Pin
Snug Fit on Shaft
3/4"
3/4"
7/8"
1/2"
1 1/8"
1 3/4"
3/4
1 1/4"
Front View
2 3/4"
2"
2 3/4"
7 1/2"

Fig. 2.—Detail of Hammer Design.

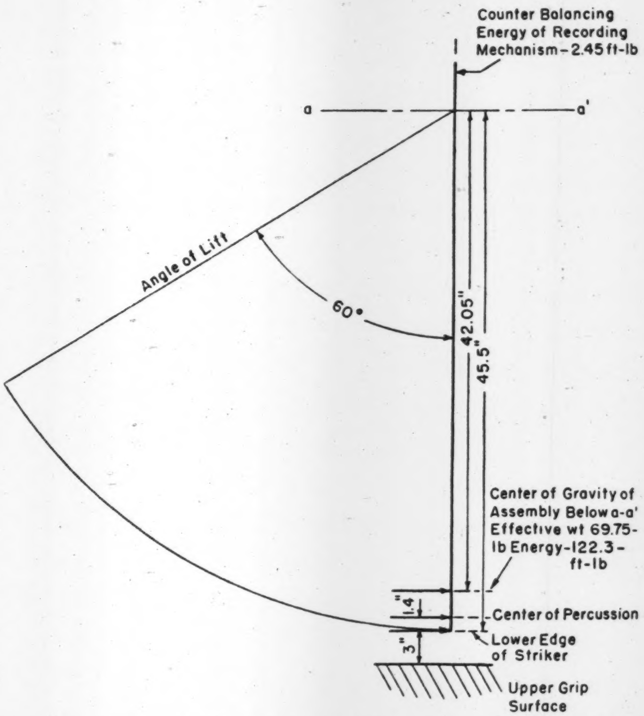
Counter Balancing Energy of Recording Mechanism - 2.45 ft-lb
a
a'
Angle of Lift
60°
42.05"
45.5"
Center of Gravity of Assembly Below a-a' Effective wt 69.75-lb Energy-122.3-ft-lb
1.4"
Center of Percussion
3"
Lower Edge of Striker
Upper Grip Surface

FIG. 3.—Schematic Diagram of Dimensions and Energy Distribution of New Pendulum.

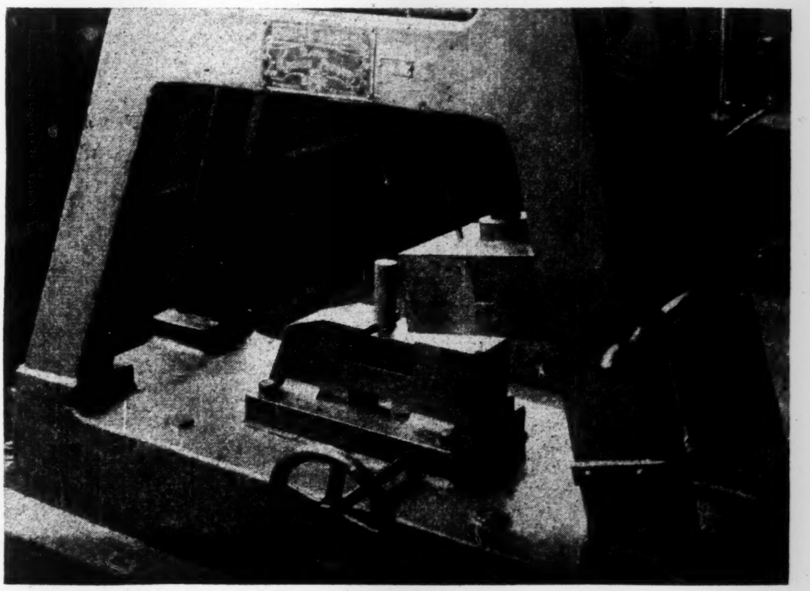

FIG. 4.—Arbitration Bar Izod Hammer and Grips.

lum shaft and a different hammer and grip arrangement designed to accommodate the standard 1.2-in. cast-iron arbitration bar in the unmachined and notchless condition. The pertinent details of the hammer and specimen grips, the principal constants of the pendulum and a photograph of the machine with a specimen ready for test are shown in Figs. 1 to 4.

The new pendulum was designed to have an energy of 120 ft-lb, the same as that of the pendulum used for testing steel. Consequently, both pendulums record impact energy on the same scale and are interchangeable without disturbing the measuring system.

The hammer is made of nickel-chromium cast iron with a heat-treated steel striker insert. The grips are steel heat treated to Rockwell C scale of 30. The pendulum length and hammer are designed to strike the specimen 3 in. above the grip surface. This dimension was set high enough to obtain ample clearance and thus assure that there would be no possibility of jamming the broken end of the specimen between the hammer and the grip. The ratio of striking height to specimen diameter roughly approximates the proportions existing in the standard Izod test for steel.

Another important design detail is that the surface of the grip on the tension side of the bar is $\frac{1}{4}$ in. lower than that on the compression side. The impact fracture tends to occur slightly below the compression grip surface, thus a lower grip on the tension side assures that the broken end will be freely swept away.

The test specimen consists of a section of an arbitration bar that may vary from 6 in. to $8\frac{3}{4}$ in. in length. No machining is required, a feature particularly advantageous with white cast irons.

Calibration of the Pendulum:

The energy of the new pendulum is readily calculated from the dimensions and weight shown schematically in Fig. 3. The weight of the entire pendulum including the hammer, shaft, guide rods, and catch is 69.75 lb. The center of gravity of this assembly was determined physically and found to be 42.05 in. below the axis of rotation. Since the angle of lift of the hammer is 60 deg, the energy stored in the pendulum is:

$$69.75 \text{ lb} \times \frac{42.05}{12} \text{ ft } (1\text{-cos } 60 \text{ deg}) = 122.3 \text{ ft-lb}$$

However, the counterbalancing energy of the recording mechanism including the shaft and pointer guide above the axis of rotation was calculated to impart a negative energy of 2.45 ft-lb.

The net potential energy delivered by the pendulum is then:

$$122.3 - 2.45 \text{ ft-lb} = 119.85 \text{ ft-lb}$$

The center of percussion was obtained from data on the period of oscillation by means of the relation:

$$T = 2\pi \sqrt{\frac{L}{g}}$$

where:

T = time in seconds for one complete swing,

L = distance in feet from the axis to the center of percussion, and

g = 32.16 ft per sec per sec.

T was found to be 2.13 sec, which meant that L was 44.1 in. and the center of percussion was 1.4 in. above the striker edge.

Similar measurements on the standard pendulum revealed that its energy was 119.6 ft-lb and that the center of percussion was 1 in. above the striker edge. Thus, in these respects, the new hammer and pendulum are quite similar to the standard instrument.

Another important feature in connection with an impact hammer is that the moments of mass forward of the striker should be equivalent to the moments of mass behind it so that there will be no tendency for rotation when striking a specimen. The new hammer fulfills this requirement, which can be determined by calculation from the dimensions in Fig. 2.

Since the specimen is a fairly bulky one, it raises the question of how much energy is required to accelerate the broken end after fracture. Experimental evidence indicates that 2 ft-lb are consumed in this way, which is an appreciable quantity in irons of low impact resistance. However, no correction is made for this factor, and the results reported include both the energy required for fracture and that required for acceleration.

ILLUSTRATIVE METALLURGICAL DATA

The mechanical properties of several grades of gray iron, martensitic white irons, and the corrosion-resistant austenitic irons are given in Table I. Where it was possible, the data were arranged to show the relation between impact resistance and some compositional variable, as well as the influence of inoculation on several compositions. A convenient term, the A. B. Izod, has been used in this laboratory to distinguish between the arbitration bar Izod test and other Izod measurements. The impact results are so designated in Table I.

In the plain gray irons, the toughness varied with carbon content, as might be expected, and decreased from 40 ft-lb at 2.3 per cent carbon to 20 ft-lb at 3.7 per cent carbon. Inoculation greatly improved the impact resistance of low-carbon iron, which is most susceptible to the formation of dendritic graphite. The network formed by phosphorus embrittles cast iron, and this fact was readily developed by the impact test.

The nickel-molybdenum irons provide interesting impact results, which reflect a change in structure not always measured by the other mechanical tests. At low alloy levels, these irons have pearlitic structures and impact resistance of 35 to 50 ft-lb. With increasing alloy content, acicular structures are developed which produce impact strengths of 60 to 90 ft-lb. The acicular irons provide the highest toughness available in gray iron, aside from austenitic material, and the sharp rise in toughness with the change in microstructure is detected only by the impact test. Other properties, except resistance to notched fatigue which is also markedly improved, simply show a gradual rise with increasing alloy level.

Detailed impact data are given for the martensitic white irons to show the normal scatter of results obtained with this material. The scatter is greater, of course, than that encountered with gray iron because of the greater notch sensitivity of this hard iron. The impact value is affected by the quality of the bar in this test. Such factors as surface and subsurface imperfections and internal voids reduce the impact strength. However, two or three tests on one composition are generally adequate to establish its impact level. The influence of the carbon content and the difference between sand and chill casting are readily apparent in the martensitic irons in spite of the moderate scatter of some of the individual results.

The austenitic irons are the toughest of the flake graphite irons and, under certain conditions of production and composition, they exceed the capacity of the machine. Inoculated Ni-Resist made with 75 per cent steel in the charge is usually not broken by the first blow of the pendulum and requires a second blow for fracture. In such cases, the impact resistance value is expressed as the number of blows without fracture plus the energy absorbed on the succeeding blow causing failure. It has been found

TABLE I.—TYPICAL IMPACT RESULTS ON VARIOUS TYPES OF CAST IRON.

	Composition, per cent									Inoculation, per cent	A.B. Izod, ft-lb	Transverse[a]		Tensile Strength, psi	BHN	Remarks
	TC	Si	Mn	Ni	Cr	Mo	Cu	S	P			Load, lb	Deflection, in.			
									PLAIN GRAY IRON							
No. 1...	2.35	2.26	0.66	....	....	....	....	0.10	(0.1)[c]	None	19	4035	0.09	47 300	262	
No. 2...	2.26	2.69	0.66	0.82	....	....	....	....	....	1 Ni 30Si	42	6595	0.17	67 800	255	
No. 3...	2.76	1.99	0.61	....	....	....	....	(0.1)	(0.1)	None	21	3800	0.10	36 100	208	
No. 4...	2.76	(2.3)	0.61	(0.7)	....	....	....	(0.1)	(0.1)	1 Ni 30Si	32	4985	0.19	53 100	212	
No. 5...	2.93	1.92	1.0	....	....	....	....	0.1	0.04	None	22	4210	0.13	43 700	235	
No. 6...	2.93	(2.3)	1.0	....	....	....	....	0.1	0.04	0.5 Fe 85Si	40	5110	0.21	50 300	228	
No. 7...	3.01	1.66	0.75	....	....	....	....	0.10	0.10	None	16	3690	0.10	40 000	228	
No. 8...	3.01	(1.9)	0.75	....	....	....	....	0.10	0.10	0.4 Fe 85Si	36	4880	0.17	45 300	215	
No. 9...	3.01	(1.8)	0.75	....	....	....	....	0.10	0.10	0.5 Ni 30Si	41	5160	0.18	51 700	223	
No. 10...	3.32	1.85	0.72	....	....	....	....	0.09	0.17	None	30	4010	0.16	36 100	203	
No. 11...	3.40	2.54	(1.0)	....	....	....	....	(0.1)	(0.1)	None	25	3630	0.17	30 100	187	
No. 12...	3.78	2.42	(1.0)	....	....	....	....	(0.1)	(0.1)	None	21	2470	0.17	21 600	158	
									EFFECT OF PHOSPHORUS							
No. 13...	3.67	2.63	0.57	....	1.14	....	....	(0.08)	0.03	None	20	3100	0.13	......	228	
No. 14...	3.67	2.63	0.57	....	1.14	....	....	(0.08)	0.20	None	20	3200	0.13	......	244	
No. 15...	3.67	2.63	0.57	....	1.14	....	....	(0.08)	0.36	None	16	3200	0.12	......	255	
No. 16...	3.67	2.63	0.57	....	1.14	....	....	(0.08)	0.53	None	11	2950	0.09	......	268	
									NICKEL CHROMIUM IRON							
No. 17...	2.95	2.0	0.8	2.25	0.75	....	....	....	....	0.5 Fe85Si	37	2790[b]	0.24	51 600	254	
No. 18...	3.14	1.56	0.66	2.24	0.62	....	....	....	....	None	37	3680	0.13	37 750	235	
No. 19...	3.22	1.93	(0.6)	1.97	0.75	....	....	....	....	None	37	4380	0.14	41 000	241	
No. 20...	3.29	1.47	0.42	1.44	0.85	....	....	....	....	None	32	4000	0.14	32 500	269	
No. 21...	3.44	1.55	0.63	2.22	0.98	....	....	....	....	None	30	3890	0.13	39 300	228	
									NICKEL MOLYBDENUM IRON							
No. 22...	3.04	2.07	0.45	2.03	....	0.34	....	....	....	1 Fe 85Si	39	5900	0.16	54 700	255	Pearlitic
No. 23...	3.06	2.17	0.62	1.70	....	0.62	....	....	....	1 Fe 85Si	78	6200	0.21	52 600		Acicular
No. 24...	2.75	2.45	0.8	1.65	....	0.75	....	....	....	0.75 Fe 85Si	71	4500[b]	0.53	67 000	286	Acicular. 30 per cent steel in charge
No. 25...	2.75	2.20	0.8	2.0	....	0.70	....	....	....	0.80 Fe 85Si	90	4590[b]	0.34	98 800	402	Acicular. 70 per cent steel in charge; tempered 5 hr at 600 F

NICKEL CHROMIUM WHITE IRON Sand Cast																
No. 26...	2.95	0.52	0.46	4.75	1.70	...	....	0.048	(0.06)	None	31, 26, 27	4650	0.11		502	....
No. 27...	3.22	0.59	0.43	4.76	1.67	....	....	0.036	(0.06)	None	26, 22, 28	4350	0.11		520	....
No. 28...	3.43	0.50	....	4.61	1.29	....	....	....	....	None	25	4120	0.11		582	....
No. 29...	3.60	0.43	0.47	4.68	1.63	....	....	0.038	....	None	27, 23	4700	0.14		606	....
Chill Cast																
No. 30...	2.95	0.86	0.46	4.75	1.70	....	....	0.048	(0.06)	None	58, 58	5640	0.10		481	....
No. 31...	3.16	0.96	(0.4)	4.60	1.44	....	....	....	....	None	33, 36, 35, 33	6300	0.10		701	Bar slowly cooled after casting
No. 32...	3.35	0.87	0.47	4.68	1.63	....	....	....	....	None	26, 31, 36, 31	6400	0.12		650	....
No. 33...	3.43	0.50	....	4.61	1.29	....	....	....	....	None	38, 44	6400	0.12		652	....
AUSTENITIC IRON																
No. 34...	2.65	2.50	1.18	13.97	2.10	....	5.95	....	....	None	63	3270	0.20	25 500	147	All scrap charges
No. 35...	2.95	1.89	(1.2)	(14.0)	(2.0)	....	(6.0)	....	....	None	85	3040	0.26	28 900	150	All scrap charges
No. 36...	2.95	2.13	1.65	14.33	2.06	....	6.33	....	....	0.75 Si[d]	95	3500	0.29	26 600	150	All scrap charges
No. 37...	2.85	1.81	(1.2)	(14.0)	(2.0)	....	(6.0)	....	....	1.2 Si	110	3225	0.39	31 500	150	All scrap charges
No. 38...	2.37	2.62	(1.2)	(14.0)	(2.0)	....	(6.0)	....	....	0.75 Si	1 Blow + 80	4650	0.58	34 600	166	75 per cent steel in charge
No. 39...	2.47	1.78	(1.2)	(14.0)	(2.0)	....	(6.0)	....	....	1.2 Si	1 Blow + 100	4820	0.60	36 000	171	75 per cent steel in charge
No. 40...	2.80	1.68	(1.2)	(14.0)	(2.0)	....	(6.0)	....	....	0.70 Si	1 Blow + 90	4060	0.56	37 180	162	75 per cent steel in charge
No. 41...	2.80	2.0	(1.2)	(14.0)	(2.0)	....	(6.0)	....	....	0.70 Si	1 Blow + 35	4060	0.48	36 860	165	75 per cent steel in charge
No. 42...	2.05	2.37	(1.2)	(30.0)	2.38	....	....	....	....	1.0 Si	1 Blow + 79	2910[b]	1.50	36 000	128	....
No. 43...	2.05	2.37	(1.2)	(30.0)	3.25	....	....	....	....	1.0 Si	1 Blow + 72	3060[b]	1.20	39 800	142	....
No. 44...	2.52	1.53	(1.2)	(30.0)	2.24	....	....	....	....	1.0 Si	100	2750[b]	1.50	30 900	117	....
No. 45...	2.52	1.53	(1.2)	(30.0)	3.23	....	....	....	....	1.0 Si	1 Blow + 44	2900[b]	0.93	35 800	130	....
No. 46...	2.81	1.45	(1.2)	(30.0)	2.32	....	....	....	....	1.0 Si	95	2085[b]	0.89	24 300	109	....
No. 47...	2.81	1.45	(1.2)	(30.0)	2.71	....	....	....	....	1.0 Si	105	2470[b]	1.13	28 850	122	....

[a] Transverse tests made on 12-in. span, except where indicated.

[b] 18-in. span.

[c] Parentheses indicate estimated composition.

[d] Added as Fe 85Si.

that irons requiring two or three blows to cause failure of the arbitration bar will have an impact resistance of 2 to 5 ft-lb when measured by the conventional Charpy test using V-notched specimens.

The foregoing data show that the arbitration bar Izod test provides a broad numerical system for distinguishing between the toughness of various types of cast irons. By modifying one of the conventional machines, a method is provided for extending its useful range to measure the impact resistance of material having considerably less toughness than steel.

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CONCLUSIONS

1. The test developed by modifying a standard 120 ft-lb Izod machine, as described in this paper, has proved to be a simple and convenient impact test for cast iron.

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[1] Technical Director, American Cast Iron Pipe Co., Birmingham, Ala.

[2] Tentative Methods of Impact Testing of Metallic Materials (E 23 – 47 T), 1949 Book of ASTM Standards, Part 1, p. 1287; Part 2, p. 1035.

[3] Report of Subcommittee XV, of Committee A-3 on Impact Testing, *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part I, p. 87 (1933).

The actual resilience is always greater than that, because of the curvature of the stress-strain curve. It will be noted that the correlation coefficient is 0.82, and the standard error of the estimate in this diagram is 26 per cent.

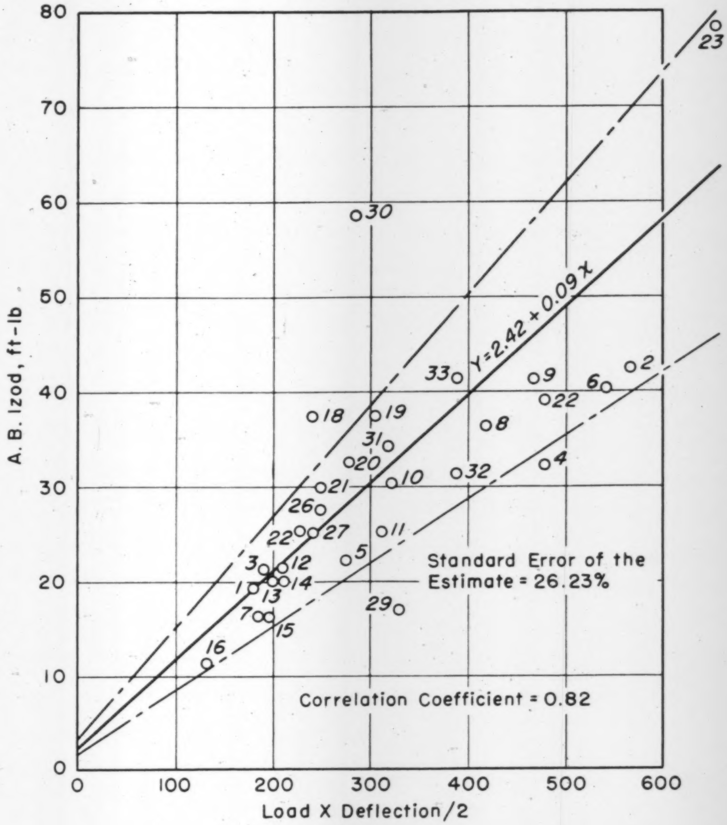

Fig. 5.

The corresponding diagram in the 1933 impact test report was of the order of 0.96 for the correlation coefficient, with a standard error of about 14 per cent, so that these results show up pretty well. Why No. 30 is so far out of line I do not know. It is one of the chill cast irons.

In Fig. 6, the same data from the authors' paper are plotted against the tensile strength cubed, divided by the Brinell hardness squared, which we found in our work on the Brinell relationships[4] to be the best ratio of these quantities.

The correlation coefficient here is 0.78 and the error of the estimate 27 per cent. A similar calculation in Fig. 13 of the above mentioned Brinell report[4] showed a correlation coefficient of 0.88 with a standard error of the estimate of 20 per cent.

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[4] J. T. MacKenzie, "TheBrinnell Hardness of Gray Cast Iron and Its Relation to Some Other Properties," *Proceedings*, Am. Soc. Testing Mats., Vol. 46, p. 1025 (1946).

THE CONSTANCY OF CALIBRATION OF ELASTIC CALIBRATING DEVICES[*]

By W. C. Aber[1] and F. M. Howell[1]

Synopsis

This paper describes the elastic load-calibrating devices used for calibrating testing machines throughout the Aluminum Company of America and discusses the results of the calibrations of the devices by the National Bureau of Standards. Since 1933, when dead-weight equipment for the work became available at the Bureau, two Amsler calibrating boxes have each been calibrated nine times by that institution. During that time the calibration factors for the devices have changed gradually but at the small rate of only about 0.1 per cent for each three years of service. Two loop dynamometers, the deflections of which are measured with dial indicators, have remained practically constant since they were first constructed and calibrated in 1944. The importance of using dial indicators that will repeat readings within narrow limits is demonstrated.

For more than thirty years the Aluminum Company of America has periodically calibrated the testing machines used for regular inspection tension tests of its products, as well as those used for research. The number of machines in use during this period has varied from only a few in 1919 up to a maximum of 87 in 32 laboratories throughout the country during World War II. At the present time there are more than 60 such machines, located in some 24 laboratories, that are regularly inspected and calibrated at intervals not exceeding one year. All these machines are multi-range machines and consequently there are a large number of load ranges that must be calibrated. The actual number of load ranges is 258 and their capacities range from 100 to 3,000,000 lb. In addition, there are four low-capacity machines having ten load ranges of 1 to 75 lb that are calibrated from time to time.

Calibrations of the testing machines are carried out in substantial accordance with ASTM Tentative Methods of Verification of Testing Machines (E 4 – 50 T)[2] using elastic calibrating devices and dead weights. The elastic calibrating devices used and their capacities are shown in the following tabulation:

Name of Device	Capacity	Method of Loading
Amsler box No. 1041	2 000 000 lb	Compression only
Amsler box No. 486	100 000 lb	Tension and compression
Morehouse Ring No. 247	25 000 lb	Compression only
Amsler box No. 379	20 000 lb	Tension and compression
A.C.O.A. dynamometer No. 3	3 000 kg	Compression only
A.C.O.A. dynamometer No. 3	2 000 lb	Compression only
A.C.O.A. dynamometer No. 4	2 000 lb	Tension and Compression

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[*] Presented at the Fifty-fourth Annual Meeting of the Society, June 18–22, 1951.

[1] Assistant Chief in Charge of Calibration Section and Chief, respectively, Mechanical Testing Division, Aluminum Research Laboratories, New Kensington, Pa.

[2] 1950 Supplement to Book of ASTM Standards, Part 1, p. 269; Part 2, p. 204; Part 3, p. 281; Part 4, p. 280; Part 5, p. 519 and Part 6, p. 263.

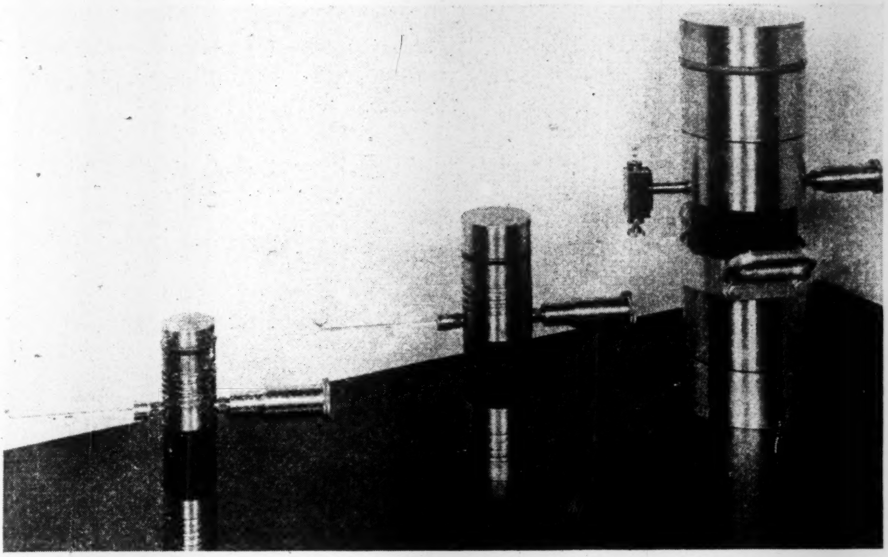

FIG. 1.—Amsler Calibrating Boxes (Left to Right) No. 379 (20,000 lb), No. 486 (100,000 lb), and No. 1041 (2,000,000 lb).

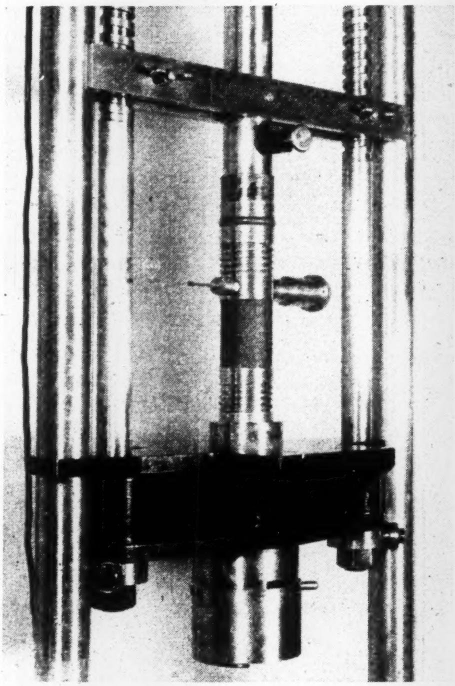

FIG. 2.—Amsler Calibrating Box No. 379 (20,000 lb) as Used in Compression.

The dead weights used are standard scale test weights of 50-lb denomination that are periodically compared with a 50-lb master weight certified by the National Bureau of Standards. Dead weights of smaller denominations are compared with a set of 13 master weights certified by the same bureau.

All the elastic calibrating devices except the Amsler of 2,000,000-lb capacity have been regularly calibrated and approved for use by the National Bureau of Standards. The reason for the exception is that the Bureau is not equipped at the present time to make direct calibrations of a device of such large capacity. This Amsler box was calibrated in a test conducted by the A. H. Emery Company and the Aluminum Company of America in cooperation with the Baldwin Locomotive Works and the National Bureau of Standards, at Eddystone, Pa., in August, 1939 (**1**).[3]

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[3] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1084.

that irons requiring two or three blows to cause failure of the arbitration bar will have an impact resistance of 2 to 5 ft-lb when measured by the conventional Charpy test using V-notched specimens.

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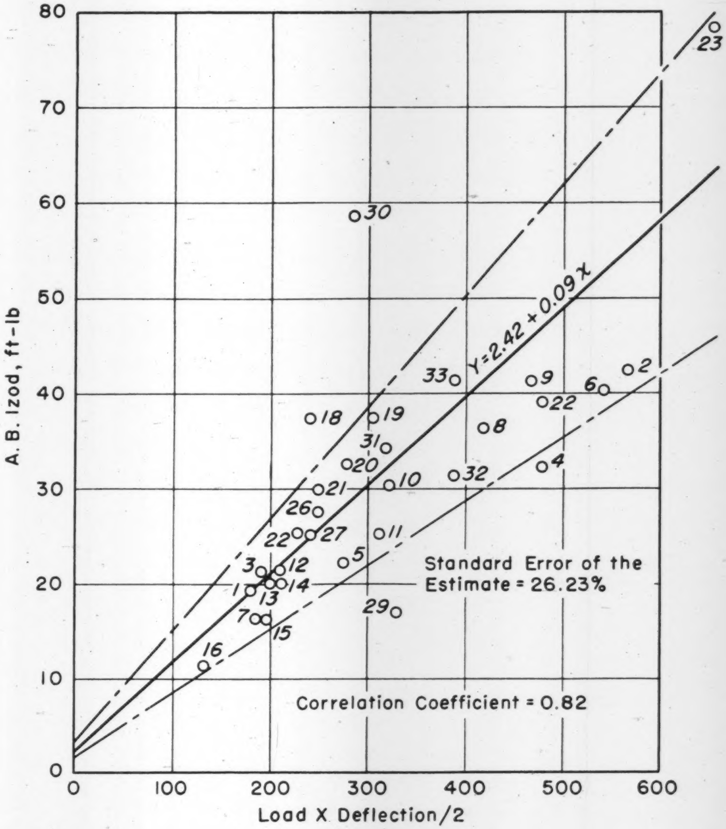

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correlation coefficient of 0.95 and the standard error of only 12 per cent.

However, since this work was on machined bars, I consider that the test proposed by the authors is a very good one.

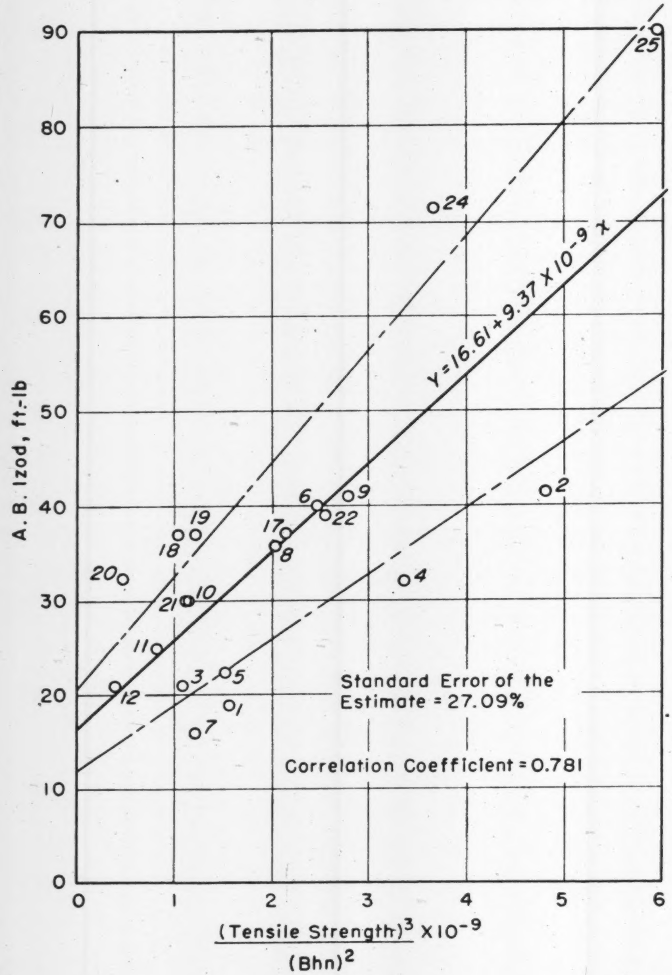

FIG. 6.

MR. A. P. GAGNEBIN (*author*).—We appreciate Mr. MacKenzie's comments very much. I think his analysis of the data that we have obtained is certainly considerably more penetrating than our own.

We used unmachined bars primarily because of their simplicity and because when produced in a core sand mold and sand blasted, they provided satisfactory test results. In the laboratory, the ability to produce the test bars is considerably greater than the facilities for machining them. In addition, the problems developed by some of the alloys in which we are interested, such as Ni-Hard, led us to the adoption of the as-cast bar.

We have tested lightly machined impact bars by the Charpy method, and at least for the cast surface quality produced in our experimental foundry, found very little difference in the spread of values between the cast surface and the lightly machined bar. However, we have not conducted tests in the Izod machine.

In view of the Impact Committee's interest in this particular test, however, we will be glad to extend our tests and make a series of tests on machined bars. We would like to have the committee's recommendation on analyses of particular interest as well as the amount of metal that should be removed.

(*author's closure*).—A comparison has been made of the A.B. Izod impact resistance of arbitration bars having as-cast and lightly machined surfaces from four different heats of cast iron. The as-cast bars had been produced in core sand molds and had good surfaces after sand blasting with diameters ranging from 1.16 to 1.20 in. The machined bars were 1.125 in. in diameter. Five or six tests were made on the four irons in each condition and the results are given in Table II. In all cases, the spread in impact values for the machined specimens was less than obtained on the as-cast bars as had been observed by Mr. MacKenzie. It is quite interesting to note that with the exception of iron No. 48, the average impact values were the same for the two surface conditions of the other irons. A microexamination of sample No. 48 showed a small amount of sub-skin porosity and graphite dendritism in the as-cast bar. This caused it to have somewhat lower A.B. Izod impact resistance than was obtained after machining off these imperfections, and the example serves to illustrate the differences encountered in testing complete castings and machined specimens.

TABLE II.—COMPARISON OF IZOD A.B. IMPACT RESISTANCE OF AS-CAST AND MACHINED CAST IRON ARBITRATION BARS.

Heat	Composition, per cent									Condition of Bars	Number of Tests	Izod AB Impact Resistance		Brinell Hardness Number
	TC	Si	Mn	S	P	Ni	Cr	Cu	Mo			Range of Values, ft-lb	Average, ft-lb	
No. 48	(3)	(2)	(0.7)	(0.06)	(0.1)	(2)				As-Cast	6	22–32	27	223
										Machined	6	31–34	33	
No. 49	3.12	1.95	(0.7)			2.33	0.58	0.18		As-Cast	6	22–34	23	255
										Machined	6	21–26	23	
No. 50	3.11	1.74	0.76			1.70			0.58	As-Cast	6	85–105	97	146
										Machined	6	85–100	95	
No. 51	2.43	2.07	(1)			14.10	2.03	6.02		As-Cast	5	1 + 36–1 + 93	1 + 61	258
										Machined	5	1 + 51–1 + 62	1 + 57	

As-cast bars 1.16 in. to 1.20 in. in diameter, molded in core sand.
Machined bars 1.12 in. in diameter, fine machine finish.
( ) Indicates estimated composition.
NOTE.—Heat No. 49 was not ladle inoculated. All others were inoculated with ferro silicon.

THE CONSTANCY OF CALIBRATION OF ELASTIC CALIBRATING DEVICES*

By W. C. Aber[1] and F. M. Howell[1]

Synopsis

This paper describes the elastic load-calibrating devices used for calibrating testing machines throughout the Aluminum Company of America and discusses the results of the calibrations of the devices by the National Bureau of Standards. Since 1933, when dead-weight equipment for the work became available at the Bureau, two Amsler calibrating boxes have each been calibrated nine times by that institution. During that time the calibration factors for the devices have changed gradually but at the small rate of only about 0.1 per cent for each three years of service. Two loop dynamometers, the deflections of which are measured with dial indicators, have remained practically constant since they were first constructed and calibrated in 1944. The importance of using dial indicators that will repeat readings within narrow limits is demonstrated.

For more than thirty years the Aluminum Company of America has periodically calibrated the testing machines used for regular inspection tension tests of its products, as well as those used for research. The number of machines in use during this period has varied from only a few in 1919 up to a maximum of 87 in 32 laboratories throughout the country during World War II. At the present time there are more than 60 such machines, located in some 24 laboratories, that are regularly inspected and calibrated at intervals not exceeding one year. All these machines are multi-range machines and consequently there are a large number of load ranges that must be calibrated. The actual number of load ranges is 258 and their capacities range from 100 to 3,000,000 lb. In addition, there are four low-capacity machines having ten load ranges of 1 to 75 lb that are calibrated from time to time.

Calibrations of the testing machines are carried out in substantial accordance with ASTM Tentative Methods of Verification of Testing Machines (E 4 - 50 T)[2] using elastic calibrating devices and dead weights. The elastic calibrating devices used and their capacities are shown in the following tabulation:

Name of Device	Capacity	Method of Loading
Amsler box No. 1041..	2 000 000 lb	Compression only
Amsler box No. 486...	100 000 lb	Tension and compression
Morehouse Ring No. 247..................	25 000 lb	Compression only
Amsler box No. 379...	20 000 lb	Tension and compression
A.C.O.A. dynamometer No. 3..........	3 000 kg	Compression only
A.C.O.A. dynamometer No. 3..........	2 000 lb	Compression only
A.C.O.A. dynamometer No. 4..........	2 000 lb	Tension and Compression

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* Presented at the Fifty-fourth Annual Meeting of the Society, June 18-22, 1951.

[1] Assistant Chief in Charge of Calibration Section and Chief, respectively, Mechanical Testing Division, Aluminum Research Laboratories, New Kensington, Pa.

[2] 1950 Supplement to Book of ASTM Standards, Part 1, p. 269; Part 2, p. 204; Part 3, p. 281; Part 4, p. 280; Part 5, p. 519 and Part 6, p. 263.

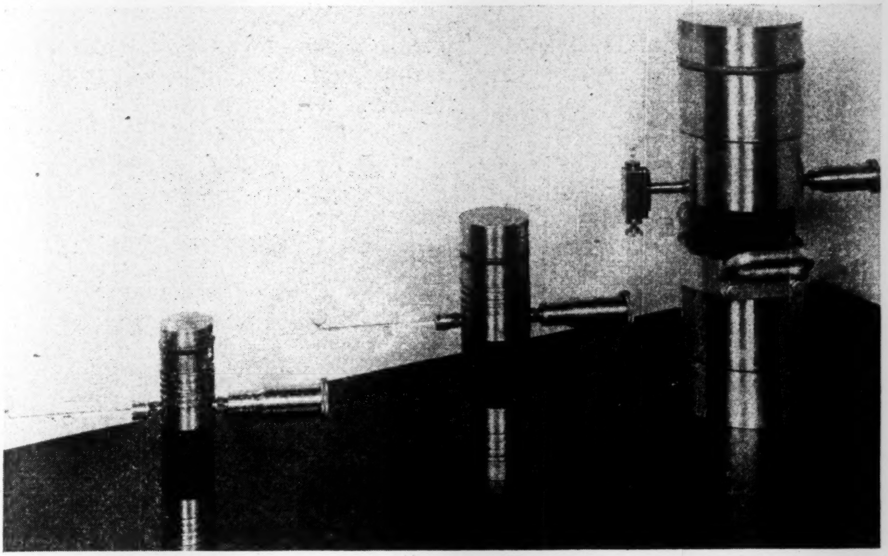

FIG. 1.—Amsler Calibrating Boxes (Left to Right) No. 379 (20,000 lb), No. 486 (100,000 lb), and No. 1041 (2,000,000 lb).

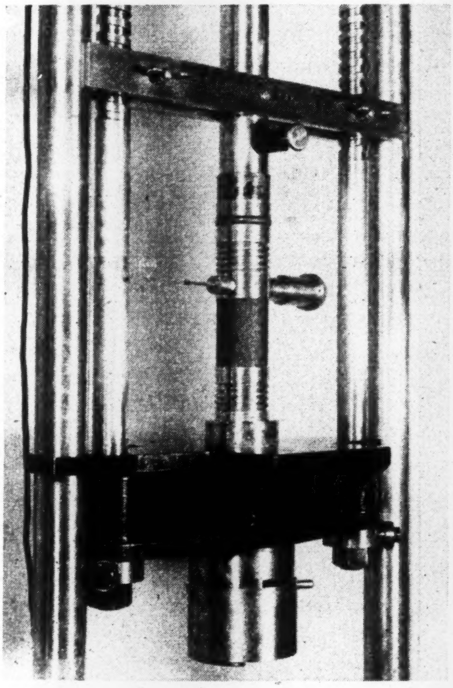

FIG. 2.—Amsler Calibrating Box No. 379 (20,000 lb) as Used in Compression.

The dead weights used are standard scale test weights of 50-lb denomination that are periodically compared with a 50-lb master weight certified by the National Bureau of Standards. Dead weights of smaller denominations are compared with a set of 13 master weights certified by the same bureau.

All the elastic calibrating devices except the Amsler of 2,000,000-lb capacity have been regularly calibrated and approved for use by the National Bureau of Standards. The reason for the exception is that the Bureau is not equipped at the present time to make direct calibrations of a device of such large capacity. This Amsler box was calibrated in a test conducted by the A. H. Emery Company and the Aluminum Company of America in cooperation with the Baldwin Locomotive Works and the National Bureau of Standards, at Eddystone, Pa., in August, 1939 (**1**).[3]

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[3] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1084.

The Amsler box of 20,000-lb capacity was put into service in 1924 using the readings for the device supplied by the manufacturer. The 100,000-lb capacity Amsler box was put into service in 1926. When the devices were purchased facilities were not available for calibrating them, but by 1933, dead-weight machines (2) for calibrating elastic calibrating devices had been installed at the National Bureau of Standards and both devices were calibrated in December of that year. The calibrations of the devices in 1933 were made at room temperature which ranged from 67 to 72 F. Later the dead-weight machines were installed in controlled-temperature rooms and all calibrations of the devices conducted after 1933 were made at a temperature of 70 F. The second calibration of the devices by means of the dead-weight machines was made in 1940 and the third was made in 1943. Additional calibrations were then made each following year.

A.C.O.A. Dynamometers of 3000-kg and 2000-lb capacity were designed and built by the Aluminum Research Laboratories in 1944. They were calibrated by the National Bureau of Standards before being put into service. They were then calibrated at least once each year through 1949.

The entire program of regular inspection and calibration of all testing machines in the various plants of the Aluminum Company of America was initiated by R. L. Templin, Assistant Director of Research and Chief Engineer of Tests, and has been carried out under his general supervision. He is also largely responsible for the design of the A.C.O.A. dynamometers that have proved so successful.

In this paper are presented the results of the several calibrations by the National Bureau of Standards of the Amsler calibrating boxes and the A.C.O.A. dynamometers. The Morehouse proving ring is not discussed because the constancy of calibrating devices of this kind is adequately covered in National Bureau of Standards Circular No. C454 (3). In 1944 Brown and Loxton (4) discussed various kinds of calibrating devices but did not cover the subject of constancy of calibration.

DESCRIPTION OF ELASTIC CALIBRATING DEVICES

The three Amsler calibrating boxes are shown in Fig. 1. The 20,000-lb capacity Amsler box is shown in Fig. 2 in position for calibrating a testing machine in compression. These devices consist essentially of hollow steel cylinders, closed at the top and bottom, completely filled with mercury. On one side of each device a capillary glass tube, terminating in a small bulb, enters the mercury chamber. Diametrically opposite the glass tube a steel tube containing a steel plunger actuated by a micrometer screw also enters the mercury chamber. These devices may be loaded in either tension or compression and when loads are applied the devices deform elastically, either increasing or decreasing the volume of the mercury chambers depending upon whether the loads applied are in tension or compression.

Under compression loading, the usual procedure is to displace mercury from the box into the capillary tube and to set the meniscus of the mercury column to an index on the capillary tube by means of the plunger. The reading of the micrometer screw with the mercury adjusted to the index on the tube and no load on the box establishes the first zero reading for the test. Load is then applied to the box and mercury is displaced from the box in proportion to the load applied. During the application of a test load to the box the micrometer screw is rotated so that when the load is fully

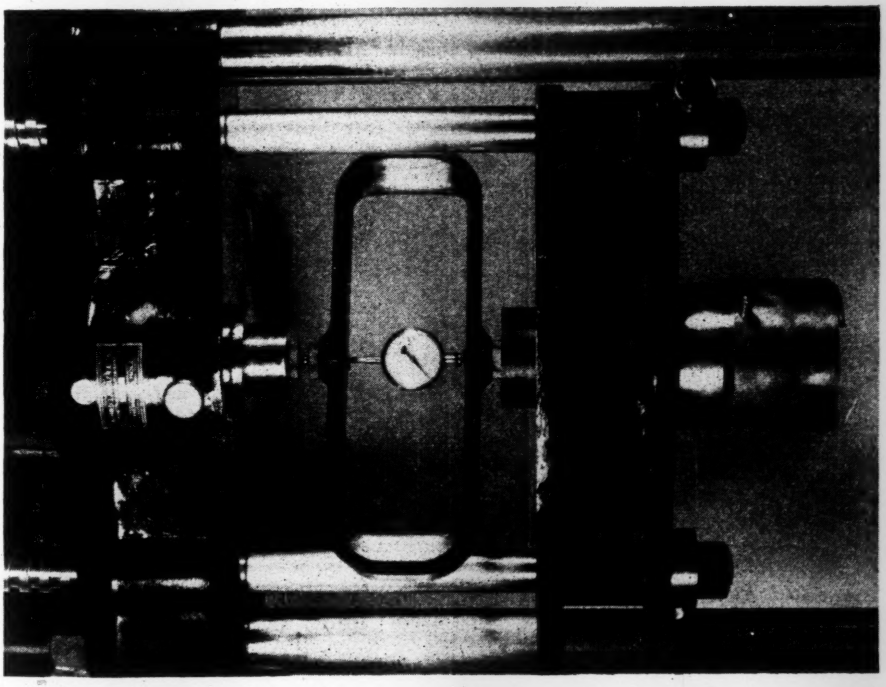


FIG. 3.—A.C.O.A. Dynamometer No. 3 (3000 kg) Used only in Compression.

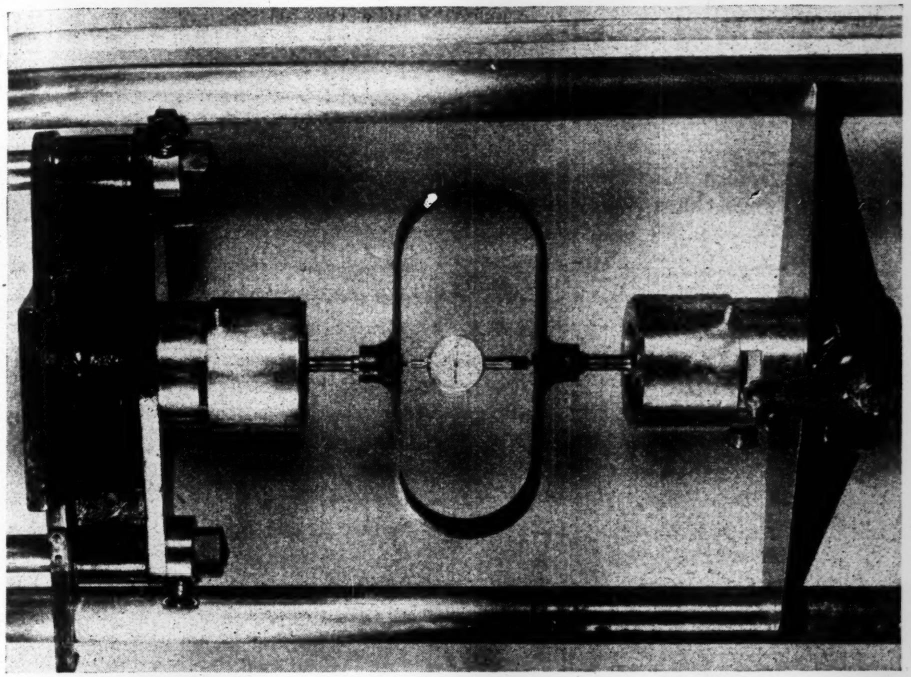


FIG. 4.—A.C.O.A. Dynamometer No. 4 (2000 lb) as Used in Tension.

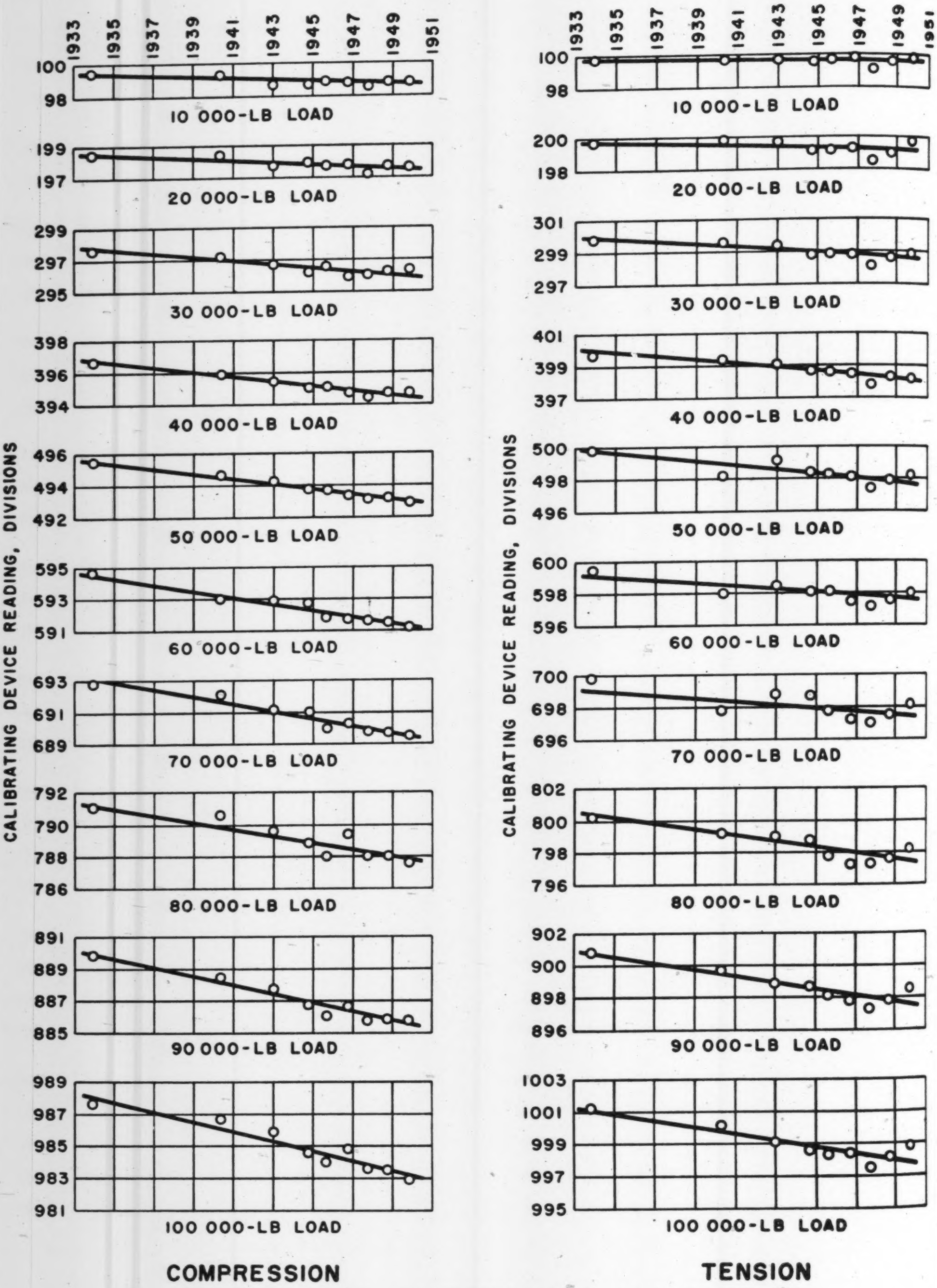
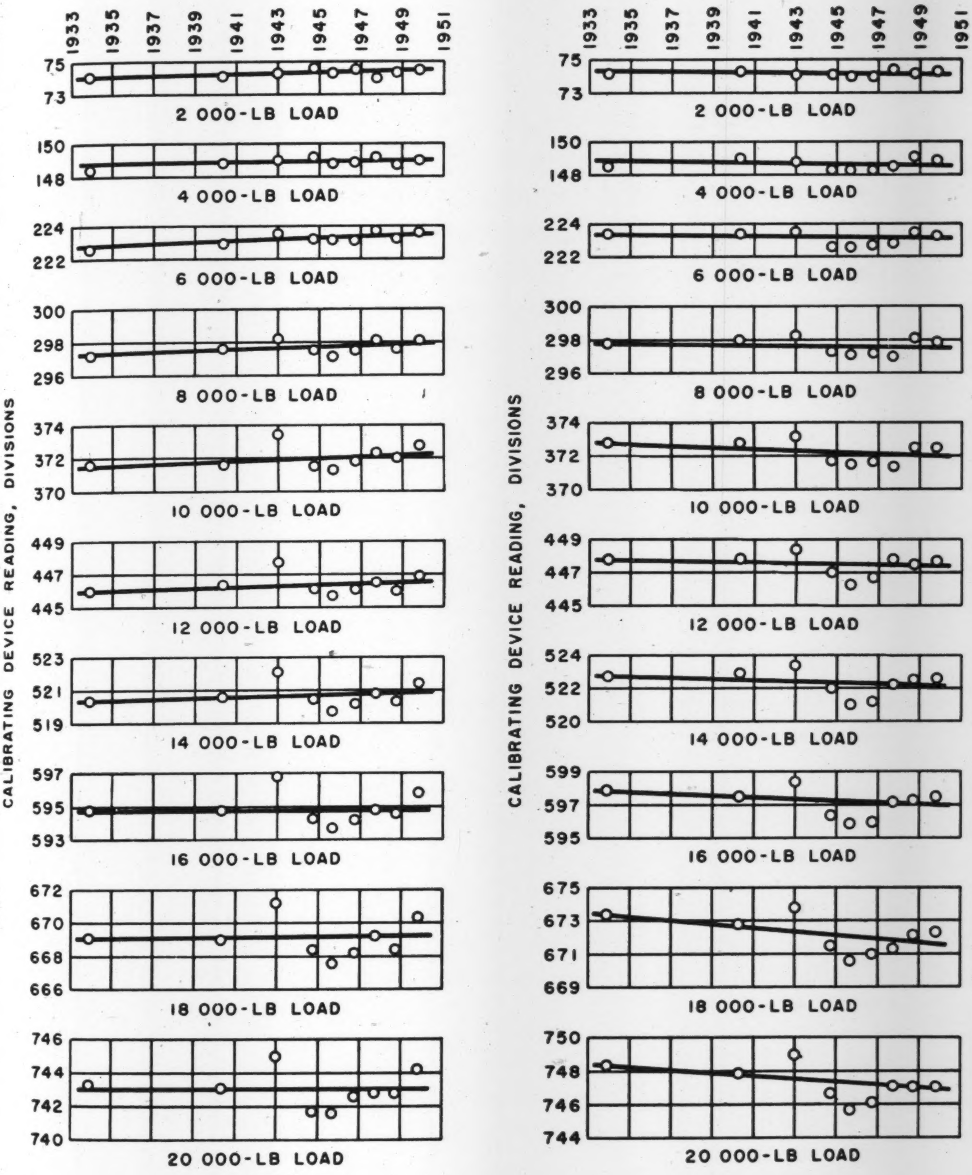


Fig. 5.—Periodic Calibrations of 100,000-lb Capacity Amsler Box. No. 486.



COMPRESSION TENSION

FIG. 6.—Periodic Calibrations of 20,000-lb Capacity Amsler Box No. 379.

applied the meniscus of the mercury column again coincides with the index and a new reading is obtained from the micrometer. After the reading under load is obtained the load is removed and a second no-load reading taken.

The difference between the load reading and the average of the two zero readings gives a value which represents the applied load. Zero readings must be obtained before and after each load reading because the readings are influenced by changes in temperature during the conduct of a test. If the temperature fluctuates rapidly satisfactory tests cannot be made but ordinary temperature changes may be compensated for by making the time interval between any load reading and the following zero reading the same as the time interval between the load reading and its previous zero reading.

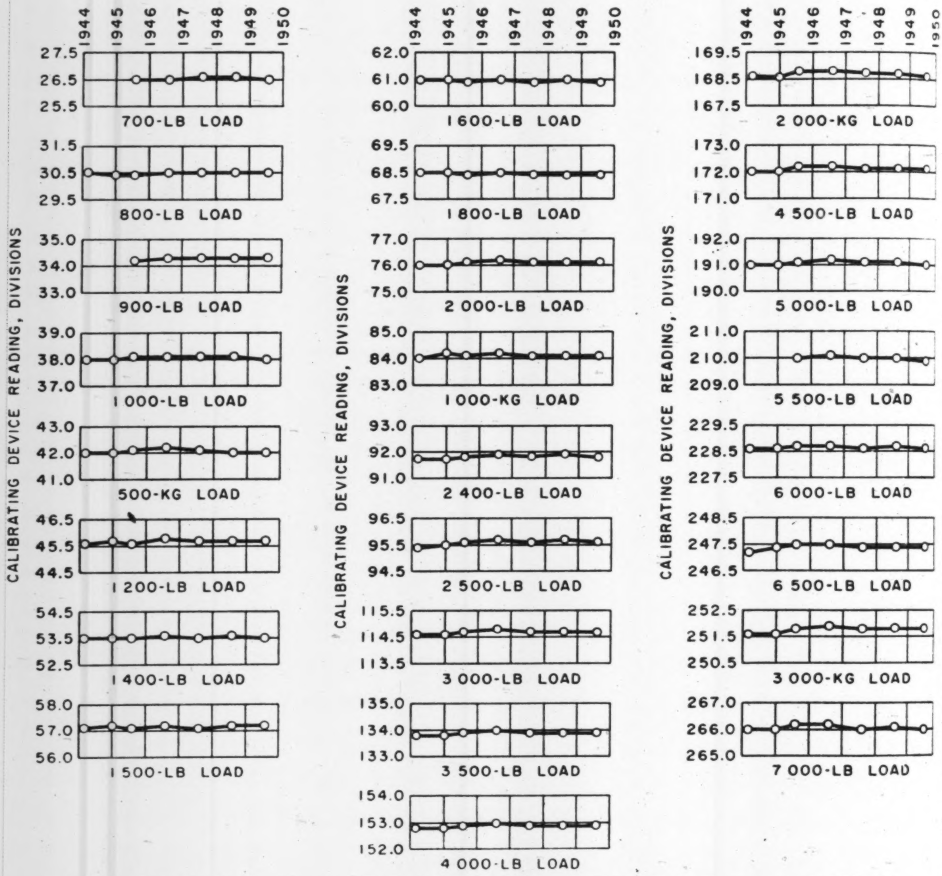


FIG. 7.—Periodic Calibrations of 3000-kg Loading Range of A.C.O.A. Dynamometer No. 3.

Under tensile loading the device functions in a similar manner except that the mercury cavity increases in volume when load is applied, and, consequently, mercury is drawn into the box and must be displaced from the box by means of the plunger as the load is applied.

The A.C.O.A. dynamometers are shown in Figs. 3 and 4. Dynamometer

No. 3 is designed for use only in compression and is equipped with two interchangeable dial indicators. One indicator (1 division = 0.001 in.) is used for loads from 700 to 7,000 lb, while the other (1 division = 0.0001 in.) is used for loads from 200 to 2000 lb. Dynamometer No. 4 is designed for use in either tension or compression, with one dial indicator for tensile loads and another for compressive loads. These devices are steel loops machined from solid forgings, designed so that dial indicators may be attached to them in such a manner that deflections of the loops can be determined for the different applied loads. The dial indicators are carefully selected but they need not have extreme accuracy. It is only necessary that the indicator readings are reproducible. An indicator reading is obtained with the dial in place and no load on the dynamometer. Load is then applied and an indicator reading is observed for that particular load. The net reading represents the applied load. There is an established net reading for each load for which the dynamometer has been calibrated and only such loads are used for calibrating testing machines. This procedure conforms to the provisions in Section 17(*c*) of ASTM Tentative Methods of Verification of Calibration Devices for Verifying Testing Machines (E 74 – 50 T).[4]

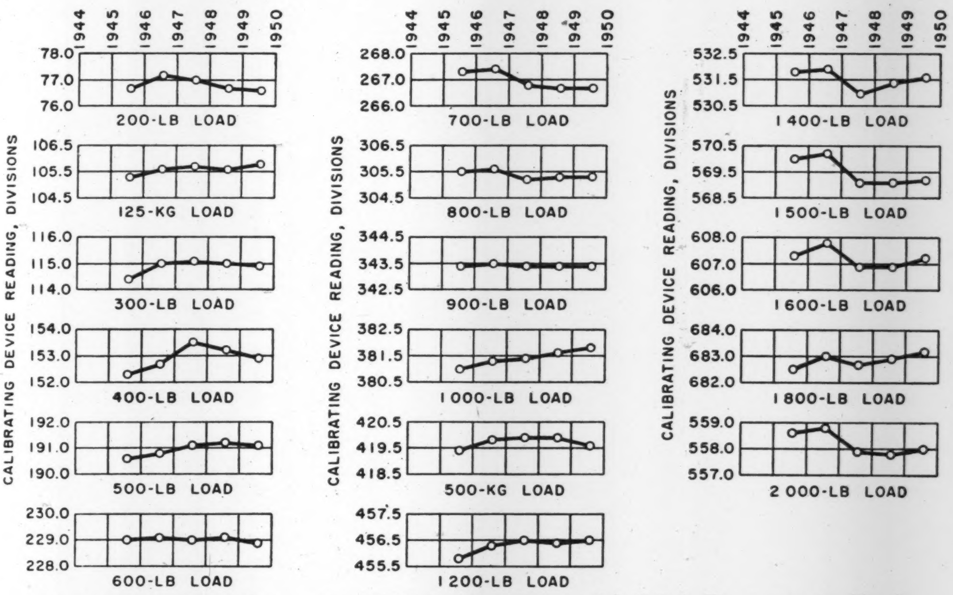


FIG. 8.—Periodic Calibrations of 2000-lb Loading Range of A.C.O.A. Dynamometer No. 3.

RESULTS OF PERIODIC CALIBRATIONS

The calibration data for the 100,000-lb capacity Amsler box are shown in Fig. 5. The calibrating device readings for each test load are plotted against the time the calibrations were made. For each load a straight line has been drawn on the chart to show the general trend of the change in readings from 1933 to 1949. Reference to these charts shows that the readings for compression loading have tended to decrease gradually, about 0.6 per cent, from 1933 to 1949. In ten-

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[4] 1950 Supplement to Book of ASTM Standards, Part 1, p. 274; Part 2, p. 209; Part 3, p. 286; Part 4, p. 285; Part 5, p. 524; Part 6, p. 268.

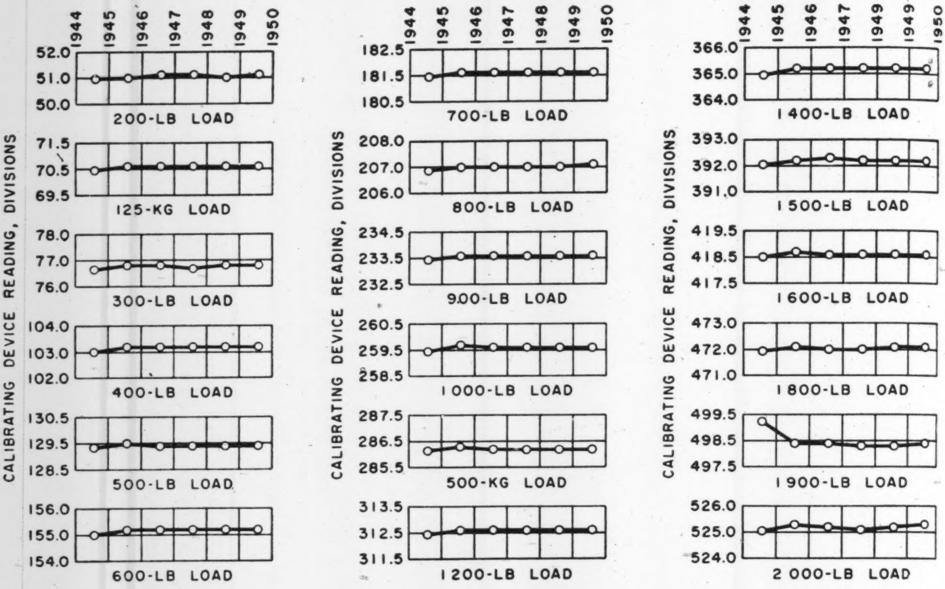

FIG. 9.—Periodic Calibrations of 2000-lb Capacity A.C.O.A. Dynamometer No. 4 in Compression.

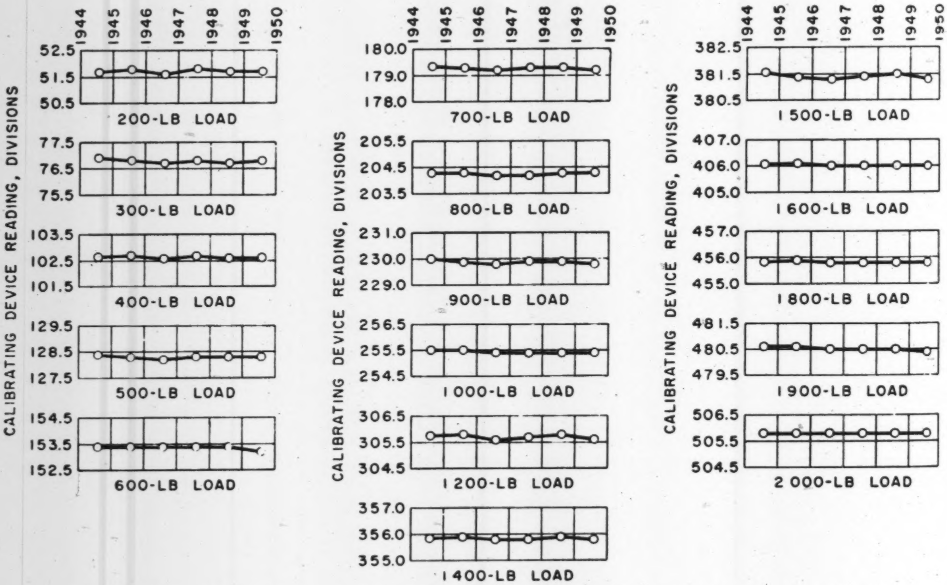

FIG. 10.—Periodic Calibrations of 2000-lb Capacity A.C.O.A. Dynamometer No. 4 in Tension.

sion they show the same gradually decreasing trend as for compression loading except that the changes are of smaller magnitude, the decrease being about 0.4 per cent.

The calibrating device readings for the 20,000-lb capacity Amsler box are shown graphically in Fig. 6. For compression loads, the readings have tended to increase about 0.2 per cent from 1933 to 1949. For tension loading throughout the same period of time the readings tended to decrease as did the readings for Amsler box No. 486 for loads in both tension and compression. The total decrease was about 0.3 per cent.

The calibration values for A.C.O.A. dynamometer No. 3 with dial indicator No. 74689 for the tests made from 1944 to 1949 are shown graphically in Fig. 7.[5] These charts show that within very narrow limits, the values obtained during that period of 6 yr have been constant with respect to time.

The calibration values for A.C.O.A. dynamometer No. 3 with dial indicator No. 927 are shown in Fig. 8. This chart shows that there is no definite trend toward increasing or decreasing values with respect to time but the values are not as uniform from year to year as the values for the same dynamometer when using dial indicator No. 74689. The difference in results may be attributed to the difference in dial indicators. The principal divisions on dial indicator 74689 represent 0.001-in. movement of its spindle and the indicator is read to 0.1 of that value. When the dynamometer is used with that indicator the no-load readings during any test are always in good agreement with each other. The principal divisions on dial No. 927 represent 0.0001-in. movement of its spindle and the indicator is read to 0.1 of that value.

The calibrating device readings reported by the National Bureau of Standards for A.C.O.A. dynamometer No. 4 with dial indicator No. 923 (compression loading) are shown in Fig. 9 and for the same dynamometer with dial indicator No. 924 (tension loading) in Fig. 10. These charts show that the values obtained for both the compression and tension loads applied to A.C.O.A. dynamometer No. 4 from 1944 to 1949 have been constant within very close limits.

DISCUSSION OF RESULTS

The results of the calibrations of the Amsler box of 100,000-lb capacity by the National Bureau of Standards from 1933 to 1949 indicate that the readings for the same applied loads have been gradually decreasing with respect to time. The average rate of indicated change is small, being about 0.03 per cent per year.

The results of the calibrations of Amsler box No. 379 of 20,000-lb capacity over the same period of time indicate that the readings for the same applied loads have been gradually increasing for the compression loads and gradually decreasing for the tension loads with respect to time. The average rate of change of readings is only about half as much as the average rate of change for Amsler box No. 486 during the same period of time. It is not apparent why the readings for Amsler box No. 379 in compression have gradually increased whereas the readings for the same device in tension and the readings for Amsler box No. 486 in both compression and tension have gradually decreased. The data indicate that the changes in calibration of the two Amsler boxes over any 3-yr period would be only about 0.1 per cent, assuming that the boxes are not mistreated or damaged in any way.

ASTM Methods E 74 – 50 T require

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[5] This dynamometer was designed for use to a maximum load of 3000 kg (6614 lb). Because it was desired to use it occasionally for a load of 7000 lb the National Bureau of Standards agreed to calibrate it for this load.

in the case of an Amsler box that a "calibration factor" be computed for each change in reading, that these calibration factors be plotted and a "calibration line" drawn through these plotted points. This calibration line is then used to evaluate a change in reading. Each calibration factor is computed as the load applied to the calibration device during its calibration divided by the change in reading of the device. The calibration line is then intended to average these calibration factors in such a way that, by using the calibration line, a calibration factor can be obtained for any change in reading within the loading range of the device.

Since the specific devices under consideration are used only at the loads for which they are calibrated and repeated calibrations have demonstrated the constancy of the change in readings, there seems to be no justification for requiring either the computation of calibration factors or the determination of a calibration line. The changes in readings can be used directly and the errors of a testing machine determined from the difference between the observed change in reading of the calibration device under the load indicated by the testing machine and the average change in reading for the exact load determined during the calibration of the device. Such a procedure would seem to provide as good a calibration of a testing machine as the much more complicated method of using a calibration line.

There seems to be no good reason, therefore, for requiring that Amsler boxes meet the "calibration line" requirement, provided they are used only at the loads for which they are calibrated. They should be acceptable for those loads for which no change in reading differs from the average change in reading for the calibration test load by more than the numerical tolerances specified. Amsler box No. 379 failed to meet this latter requirement for a load of 10 per cent of capacity ten times out of eighteen and, therefore, would not always be acceptable for such a small load. Amsler box No. 486 met this requirement in 14 out of the 18 calibrations at 10 per cent of its capacity load. For loads of 20 per cent of capacity or more both Amsler boxes met the requirement reasonably consistently, there being only 2 or 3 instances for each device when one or two readings of the three taken during one calibration differed by more than 0.25 per cent from the average.

Study of the data for A.C.O.A. dynamometer No. 3 with dial indicator No. 74689 and A.C.O.A. dynamometer No. 4 with dial indicators Nos. 923 and 924 in Figs. 7, 9, and 10, shows that the readings of these devices are relatively constant with respect to time. The differences between the maximum and minimum readings obtained for more than 80 per cent of the loads have not exceeded 0.2 division from 1944 to 1949. During the same period of time the differences between the maximum and minimum readings for the remaining loads, with one exception, have not exceeded 0.3 division. The exception applied to the reading for the 1900-lb load in compression applied to A.C.O.A. dynamometer No. 4 in 1944. That reading is one division higher than the readings obtained for the same load in 1947 and 1948 and 0.9 division higher than the readings obtained in 1945, 1946 and 1949. Study of all the data for that device leads to the conclusion that the observation in 1944 might have been in error by one division.

The readings for A.C.O.A. dynamometer No. 3 with dial No. 927 are shown in Fig. 8. There is no gradual change in readings with respect to time but the readings are not as consistent as those obtained when dial No. 74689 is used with the same dynamometer. The differences between the maximum and minimum readings for the loads ranged from 0.1 to 1.2 divisions. The actual indicated changes, however, in inches were small because each division represents 0.0001 in. The reason for these relatively large differences must be attributed to the dial indicator, which cannot be depended upon to repeat readings as closely as is desirable.

The A.C.O.A. dynamometers met the requirements of ASTM Methods E 74 - 50 T consistently for loads of 20 per cent of capacity and higher; in fact, except for dynamometer No. 3 with indicator No. 927 (1 division = 0.0001 in.), they consistently met the requirements for all loads for which the change in reading was 500 times the minimum estimated change in reading, which, for dynamometer No. 4, covers loads as low as 10 per cent of capacity.

Incidentally, the requirement in ASTM Methods E 74 - 50 T that for certain devices used only for those loads for which they are calibrated "for each change in reading the corresponding calibration factor shall be calculated and the average calibration factor shall be determined for each test load" makes it mandatory that certain calculations be made that are entirely unnecessary. The changes in readings can be averaged and each of those averages used with the same degree of accuracy without dividing each into the calibration load and then multiplying the calibration factor by the change in reading.

CONCLUSIONS

From a consideration of the results of calibrations by the National Bureau of Standards of Amsler boxes Nos. 486 and 379 from 1933 to 1949 and A.C.O.A. dynamometers Nos. 3 and 4 from 1944 to 1949, it has been concluded:

1. The readings for Amsler box No. 486 of 100,000-lb capacity for loads in both compression and tension have decreased about 0.5 per cent at a fairly uniform rate from 1933 to 1949. The average rate of decrease was about 0.03 per cent per year.

2. The readings for Amsler box No. 379 of 20,000-lb capacity for loads in compression have gradually increased and for loads in tension have gradually decreased from 1933 to 1949. The total change in both cases was about 0.25 per cent or about 0.02 per cent per year.

3. The rates of change have been such that, provided the Amsler boxes are neither damaged nor mistreated, the change over a 3-yr period would be expected to be only about 0.1 per cent.

4. The readings of A.C.O.A. dynamometer No. 3 of 3,000-kg capacity using dial indicator No. 74689 and A.C.O.A. dynamometer No. 4 of 2000-lb capacity using dial indicators Nos. 923 and 924 have been practically constant for the tests made from 1944 to 1949.

5. The readings of A.C.O.A. dynamometer No. 3 of 2000-lb capacity with dial indicator No. 927 are less consistent than is desirable. The precision of this dynamometer at low loads could be improved by a dial indicator of the proper size and range with 0.0001-in. divisions that could be depended upon to repeat readings within narrower limits.

6. The present requirements of ASTM Methods E 74 - 50 T that "calibration factors" and "calibration lines" be determined for calibrating boxes and that for devices of the Alcoa dynamometer type "calibration factors" be calculated are unnecessary to assure suitable accuracy of testing machines used for ordinary inspection testing.

REFERENCES

(1) "Calibration of Large Testing Machine," *Technical News Bulletin No. 270,* National Bureau of Standards, October, 1939, pp. 103–104.

(2) B. L. Wilson, D. R. Tate, and G. Borkowski, "Dead-Weight Machines of 111,000- and 10,100-Pound Capacities," *Circvlar C446,* Nat. Bureau Standards (1946).

(3) B. L. Wilson, D. R. Tate, and G. Borkowski, "Proving Rings for Calibrating Testing Machines," *Circular C454,* Nat. Bureau Standards (1946).

(4) G. L. Brown and H. T. Loxton, "Load Calibration Equipment for Testing Machines," *The Journal of the Institution of Engineers,* Australia, Vol. 16, pp. 235–239 (1944).

DISCUSSION

MR. D. R. TATE.[1]—This paper is of real interest, as it presents the carefully compiled history of some very well-maintained and also rather unusual devices.

The two loop dynamometers have been an outstanding success, and they feature a method of mounting the dial indicators which permits a much higher degree of repeatability than other methods of mounting.

The history of the Aluminum Company of America's two Amsler boxes is unusually complete. It represents the only complete history we have for Amsler boxes calibrated in both tension and compression. In looking back over the records of these Amsler boxes, I find one important factor that I believe the authors have omitted in the paper. That factor is the care exercised by the engineer who has maintained these devices for the Aluminum Company in such very fine condition. Mr. Aber, who is personally in charge of them, has, through the exercise of unflagging effort, skill, and patience, succeeded in keeping these two devices in very respectable working order.

It may be of interest to know that the National Bureau of Standards has a record of some 27 Amsler boxes, submitted over the past twenty or thirty years. Of these 27 devices, 14 were calibrated once and have never returned. Of the remaining few, eight have a fairly long and consistent history. This includes the two boxes owned by the Aluminum Company of America.

As a quick survey, the following comparison may give some indication of the relative constancy of other Amsler boxes now in service. The average change in capacity reading, between successive calibrations, for these eight devices was about 0.2 per cent, with two of them exhibiting, at some time or other, changes exceeding 0.75 per cent, four of them exhibiting changes at some time exceeding 0.4, and seven exhibiting some changes exceeding 0.3 per cent. This represents change between successive calibrations and not the total drift of the devices.

In considering the acceptability of a class of instruments, the performance of the class should not be judged by the performance of the best examples in the class.

MR. M. J. MANJOINE.[2]—This paper was enjoyable for two reasons.

First, it may stimulate other laboratories of the country to use calibrating devices in the checking of their testing machines.

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[1] Physicist, National Bureau of Standards, Washington, D. C.

[2] Research Engineer, Mechanics Dept., Westinghouse Research Laboratories, Westinghouse Electric Corp., East Pittsburgh, Pa.

Secondly, we at Westinghouse, realize that since the company has several testing laboratories throughout the country, our testing machines should be calibrated by the same device or to some common standard in order to obtain comparable test results. We have initiated the practice of periodically calibrating our testing machines at the research laboratories. Up to the present time, we have calibrated the machines with our own "standard." That is, we have taken a standard lever machine and calibrated the lever arm ratio, and then using known dead weights we have calibrated our elastic calibrating devices. These are similar to the ones described in the paper. However, stimulated by this paper, we are planning to send our calibrating devices to the National Bureau of Standards for a check.

I have two questions to ask. First, what is the nature of the steel that is used for these calibrating devices built by the Aluminum Company of America? They seem to be quite stable. Second, what is the manner of attaching the dial gage? This is not clear in the illustrations of the paper.

MR. T. J. DOLAN.[3]—I notice that several of the devices mentioned in this paper may be used to measure both tensile and compressive loads. We have had a little difficulty with some elastic load-measuring instruments made in our machine shop. These devices use resistance wire strain gages as the measuring element and we find that there is a little hysteresis effect when the loads are reversed. I wonder if the authors have noted any such effect in the devices that they were using. If the instrument is very carefully calibrated in compression and then immediately calibrated in tension, is any shifting of the data or change in the "zero" load reading noted?

MR. W. H. MUNSE.[4]—Having designed a few dynamometers similar to the Aluminum Company of America units, I should like to know if Mr. Aber has any information on the maximum strains which are obtained in these dynamometers?

CHAIRMAN J. R. TOWNSEND.[5]—As I understand it, the calibrating device is calibrated by the Bureau of Standards, by the use of dead weights, once each year. May I ask how often the testing machines are calibrated with calibrating devices, and whether there is a regular routine for doing this?

Then I should like to make a suggestion to the testing machine manufacturers and others who are present here. In a sense, these calibrating rings and boxes represent the acme of mechanical perfection. We are in a new age now. You might describe it as an electronics age, and I should like to leave this suggestion, that perhaps some electronic device may be developed which can be attached to a testing machine at any time, and at any point would give a calibration of the machine.

MR. W. C. ABER (*authors' closure*).—Mr. Tate's remarks are very much appreciated. As he indicates, it should be realized that calibrating devices, if they are to be expected to retain their precision, must be given good care and should not be entrusted to inexperienced personnel.

In response to the questions of Mr. Manjoine, it may be said that A.C.O.A. dynamometers Nos. 3 and 4 were machined from alloy steel forgings of the following nominal composition: carbon 0.4 to 0.5, tungsten 2.5, chromium 1.5, vanadium 0.25, manganese 0.2 and silicon 0.2 per cent. They were heat treated by quenching in oil from 1760 F and drawn at 600 F.

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[3] Research Professor of Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

[4] Research Assistant Professor of Civil Engineering, University of Illinois, Urbana, Ill.

[5] Materials Engineer, Bell Telephone Laboratories, Inc., Murray Hill, N. J.

The dial indicators are not permanently attached to the devices but are arranged so that they may be placed in position when needed and then removed and stored separately when the devices are not being used. Each indicator is equipped with two hardened steel conical points which fit into holes in small steel fittings permanently attached to the inside opening of the dynamometer. One of the points is screwed into the end of the indicator spindle and the other is securely attached to a bracket mounted on the back of the indicator in such a manner that the axes of the conical points coincide with the axis of the indicator spindle. The axes of the holes in the steel fittings coincide with the axis of the dynamometer through which load is applied. The method of mounting an indicator in a dynamometer is to push the indicator spindle into the body of the indicator and then place the fixed point of the indicator into the lower hole of the dynamometer. The spindle is then permitted to move out of the body of the indicator while its point is guided into the upper hole. The indicator, if properly inserted, will stand vertically in the dynamometer supported by the fixed conical point at the bottom and by the conical point in the end of the spindle at the top. The indicator should be free to rotate about its vertical axis and have sufficient spindle movement to accommodate the maximum loads to be applied.

We are unable to answer Mr. Dolan's question completely because a different dial indicator is used for loads in tension than is used for loads in compression. Also, different shackles or adapters are used for the different conditions of loading, making it impossible to detect the presence or absence of hysteresis when changing from compression loading to tension loading.

As the devices are used, our standard practice is to preload them to the maximum load for which calibrations are to be made. After that has been done, the zero readings before and after subsequent loadings agree very nicely.

Our reply to the question by Mr. Munse is that we have not attempted to measure the maximum strains in the different devices. When they were originally designed, however, it was calculated that nominal stresses as great as 122,000 psi might be encountered.

In reply to the question of Mr. Townsend, it may be said that it is our practice to calibrate our testing machines at least once each year. More frequent calibrations are made, however, if there are any reasons for believing them to be necessary.

In closing I would like to say that both Mr. Howell and I deeply appreciate the interest displayed in our paper, especially by those who have discussed it.

REPORT ON FURTHER STUDIES TO DEVELOP AN ACCELERATED TEST PROCEDURE FOR THE DETECTION OF ADVERSELY REACTIVE CEMENT-AGGREGATE COMBINATIONS*

By Thomas E. Stanton[1]

Synopsis

Considerable additional research has been carried on during recent years supplementing the studies reported by the author in 1943 on "Studies to Develop an Accelerated Test Procedure for the Detection of Adversely Reactive Cement-Aggregate Combinations."[2]

At that time it was reported that best over-all long-time results were obtained through curing in sealed containers, but it was emphasized that this conclusion was based on tests with reactive aggregates of the type encountered in California.

Recent tests indicate that the magnitude of the reaction may be materially accelerated by increasing the percentage of mixing water to produce a flow of 140 to 150 instead of limiting the water content to. that which will produce a flow of only 100 to 115 as designated for use in ASTM Tentative Method of Test for Potential Alkali Reactivity of Cement-Aggregate Combinations (C 227 - 50 T).[3]

The tests recorded herein have been undes way for over one year, with reactive aggregates from numerous sources. They are reported herewith in support of a proposed change in the sealed container procedure which is currently under consideration.

Other tests sustain previous conclusions that in the case of cement-aggregate combinations where the excessive expansion results solely from a chemical reaction between the cement and the aggregates, constant immersion or alternate wetting and drying may act as a deterrent rather than an accelerator as compared with the sealed container method.

Recent Tests:

Observation of the performance of hundreds of laboratory specimens during the past twelve years as well as of numerous field structures has emphasized the importance of moisture in developing and sustaining the reaction between aggregates and cements.

Just as in the case of the percentage of reactive particles, there appears to be an "optimum" amount of water to produce the quickest and greatest results.

If the specimens are stored under dry conditions, no appreciable measurable reaction takes place. When stored under excess exterior moisture conditions, there is a tendency to nullify or dissipate some of the reactive processes.

In the case of structures, the exposed

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* Presented at the Fifty-fourth Annual Meeting of the Society, June 18-22, 1951.

[1] Materials and Research Engineer, California Division of Highways, Sacramento, Calif.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 43, 1943, p. 875.

[3] 1950 Supplement to Book of ASTM Standards, Part 3, p. 17.

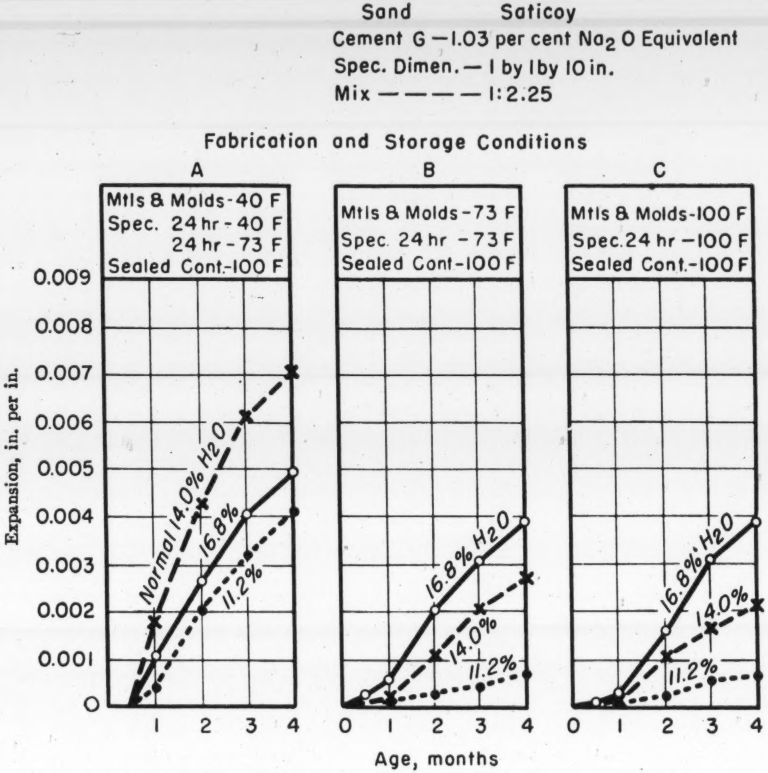


FIG. 1.—Showing Effect of Percentage of Mixing Water and Temperature on the Rate of Expansion of an Adversely Reactive Cement-Aggregate Combination. This test series started March, 1950.

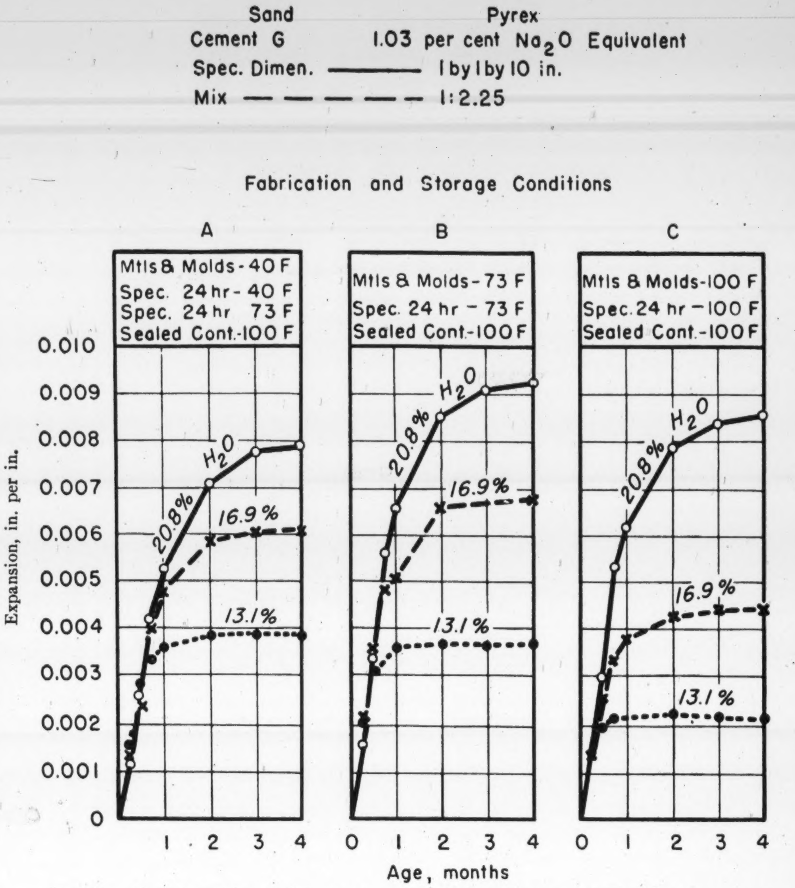
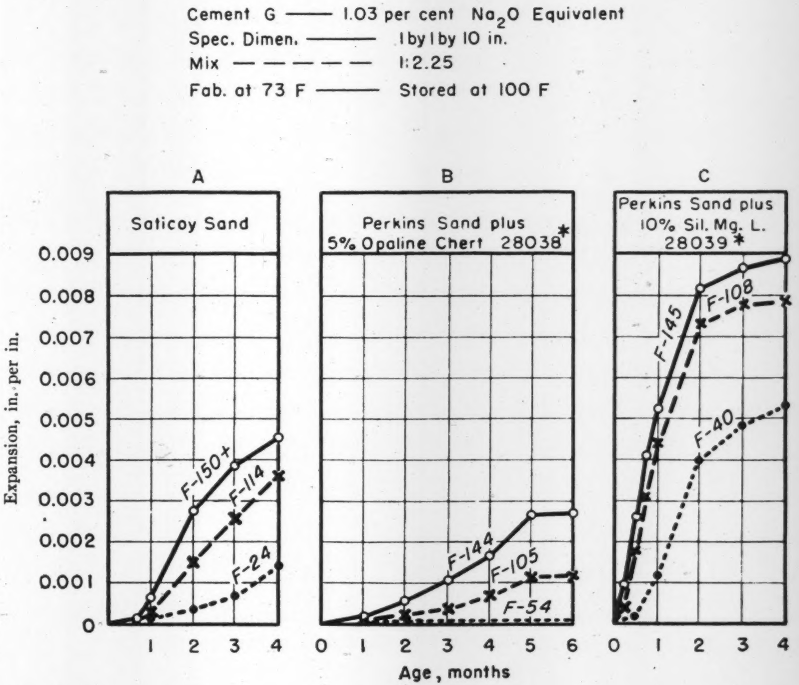


FIG. 2.—Showing Effect of Percentage of Mixing Water and Temperature on the Rate of Expansion of a Highly Reactive Cement-Pyrex Glass Combination.

portions usually show the earliest and greatest distress, whereas areas in the same structure which are protected from rain or moisture from other sources frequently develop little distress.



"F" refers to Flow.

* 5 per cent of highly reactive Opaline Chert No. 28038 is obviously in excess of the "pessimum" percentage, whereas 10 per cent of the less reactive Siliceous Magnesian Limestone No. 28039 is somewhat less than the "pessimum" percentage.

FIG. 3.—Showing Effect of Percentage of Mixing Water on the Magnitude of the Expansive Reaction with Three Reactive Aggregate Combinations.

Note close similarity of curves for Saticoy Sand (A) with "B" in Fig. 1.

Based on the above observations it was concluded that as the amount of moisture in the specimen has a material bearing on the results, the amount of mixing water normally used might be too small to produce the earliest or greatest action.

A large number of tests have been conducted during the past year in which the amount of mixing water has been varied from low to as high as practicable so as not to exceed the capacity of the flow table (150).

It was found that the earliest and usually greatest ultimate expansion resulted from a water ratio which produced a flow of 140 to 150 as compared with the ratio designated for use in ASTM method C 227 which specifies that the amount of mixing water shall be such as to produce a flow of between 100 and 115. Sometimes the acceleration with the higher ratio is very marked.

Sustaining Test Data:

Figures 1 to 11 illustrate the results of the recent studies on the effects of different percentages of mixing water. For the most part, the titles to the figures are self-explanatory.

However, a few additional comments

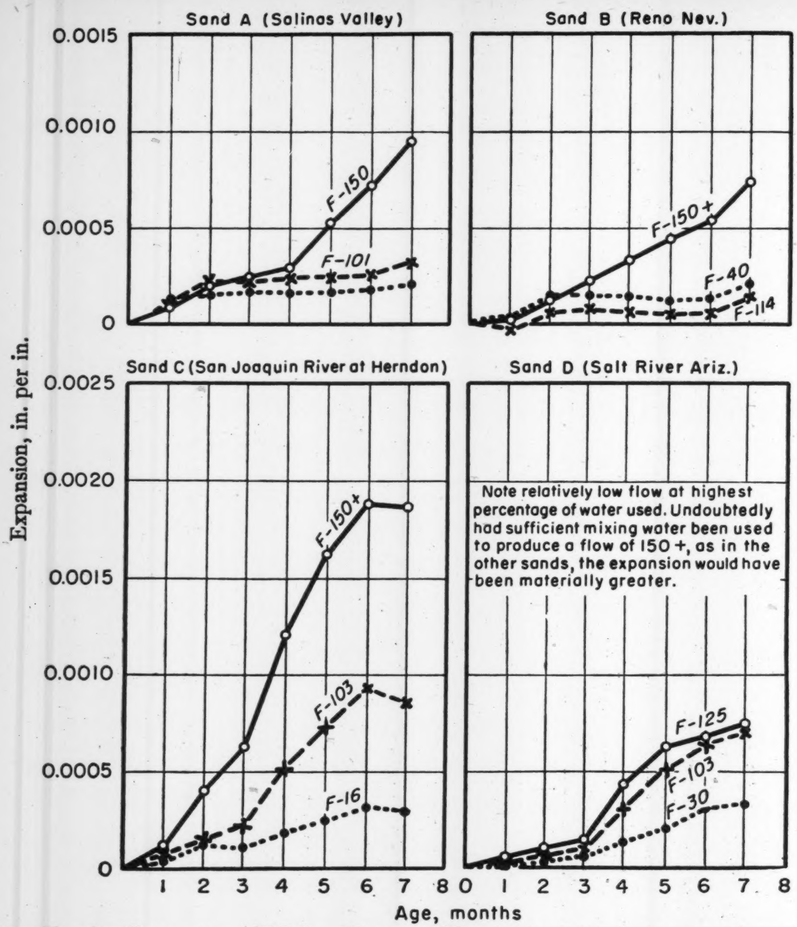


Fig. 4.—Showing Percentage of Mixing Water on Magnitude of Expansive Reaction with Four Commercial Sands Normally Considered Mildly Reactive.

Cement G 1.03 per cent Na_2O Equivalent. Spec. Dimen. 1 by 1 by 10 in. Mix 1:2.25. Fabricated at 73 F. Stored at 100 F.

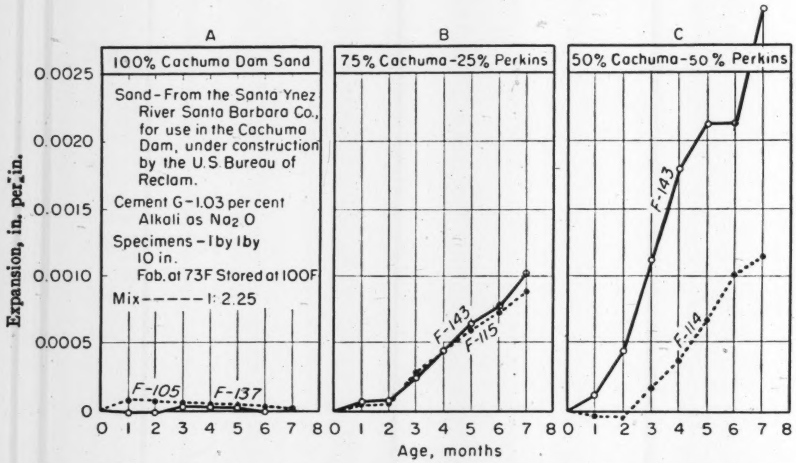


Fig. 5.—Tests to Determine if the Failure of a Potentially Reactive Sand to Develop Appreciable Expansion, in Combination with a High-Alkali Cement, Might Not be Due to an Excess of Reactive Particles, Substantially Beyond "Pessimum." Tests were made with high as well as normal percentages of mixing water.

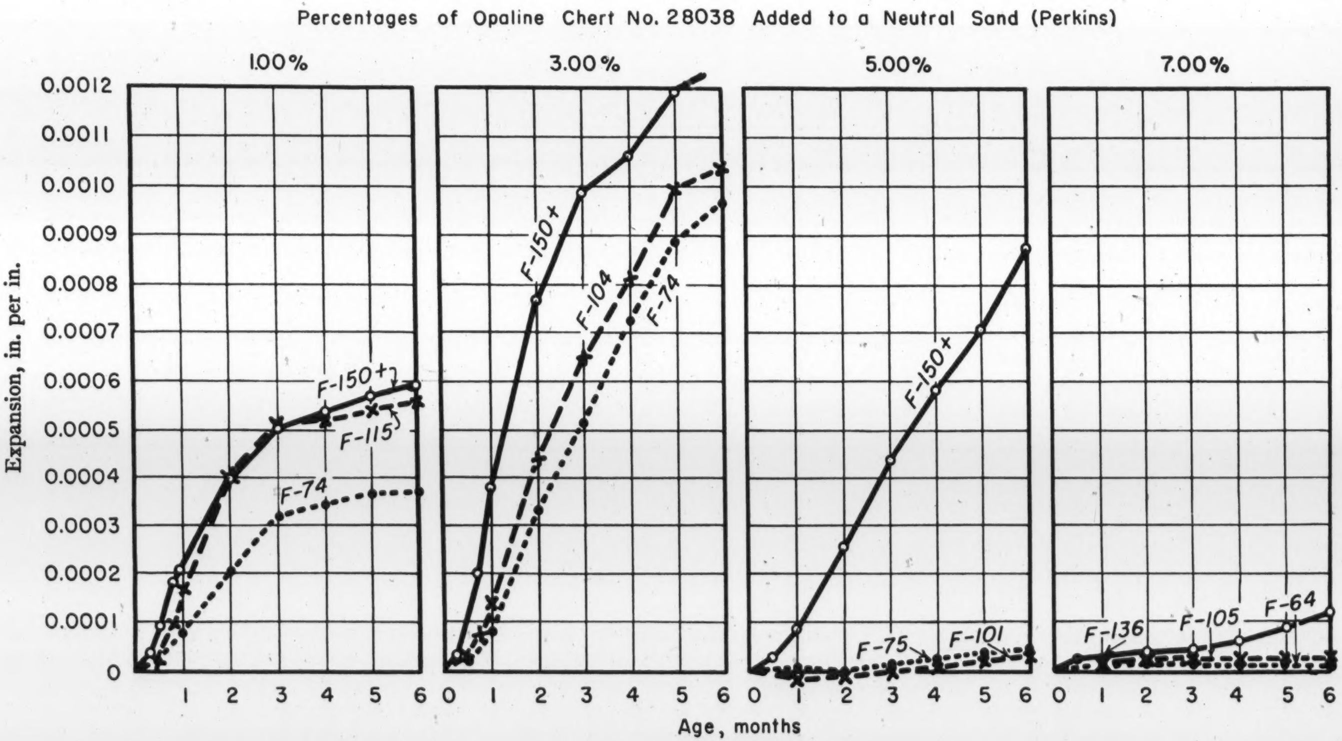


FIG. 6.—Tests to Determine Effect of Various Percentages of Mixing Water in Highly Reactive Aggregate High-Alkali Cement Combinations Containing Different Percentages of the Sand Reactive Ingredient (Opaline Chert No. 28038).

Note the "Pessimum" percentage (3.00) and high expansion developed through high mixing water in the case of the 5.00 per cent addition even through minor expansion developed in six months with the normal percentage of water.

are in order. The first studies (the results of which are illustrated in Figs. 1 and 2) were conducted at different fabrication and initial curing temperatures ranging from 40 F to 100 F.

While fabrication and initial curing at 40 F appeared to accelerate the results in some cases, the evidence is not sufficiently consistent or conclusive to justify working under the more difficult procedure required to bring all ingredients to 40 F and hold at that temperature for the first 24 hr.

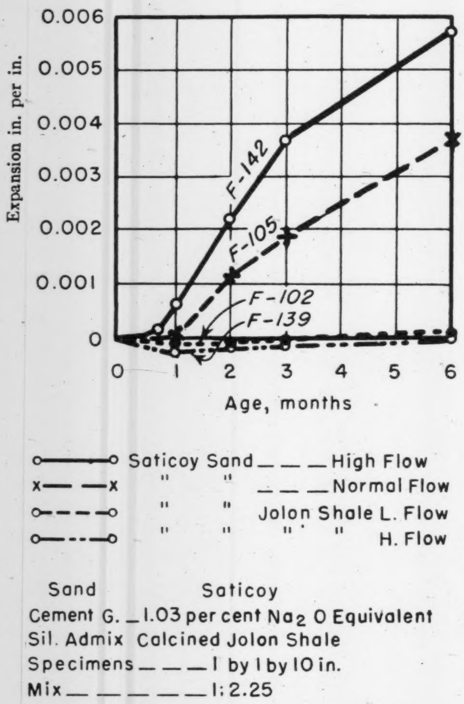


FIG. 7.—Tests to Determine the Comparative Corrective Effects of 10 per cent Pulverized Jolon (Monterey Type) Shale as a Substitution for an Equivalent Percentage of the Cement, with Normal and High Percentages of Mixing Water.

Result with uncalcined shale identical with calcined product.

Nor does there appear to be any advantage in fabricating at 100 F (Figs. 1 and 2).

Therefore, insofar as temperature is concerned, there appears to be no advantage in departing from current standard procedure, namely, fabricate and cure first 24 hr at 73 ± 3 F and then store in a sealed container at 100 F.

However, with regard to the percentage of mixing water, there is a decided advantage in using the greatest quantity that will produce a flow up to the capacity of the flow table at least.

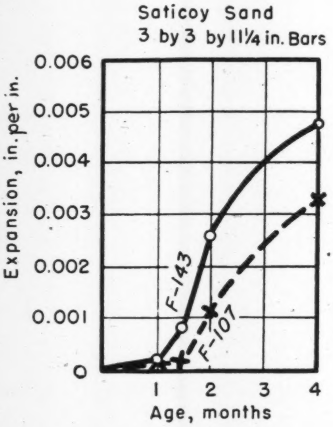


FIG. 8.—Comparing Effect of High and Normal Percentage of Mixing Water with 3 by 3 by 11¼-in. Bars of Saticoy Sand and Cement G—1.03 per cent Na_2O Equivalent.

This conclusion is sustained through the tests which are illustrated in the various figures.

Monterey Shale as a Corrective:

The corrective effects of 10 per cent of the true Monterey shale, regardless of the percentage of mixing water, are well illustrated in Fig. 7. It will be noted that with the highly reactive Saticoy commercial sand, the expansions up to six months were negligible regardless of the percentage of mixing water when 10 per cent of the Jolon shale was substituted for an equivalent per cent of the cement.

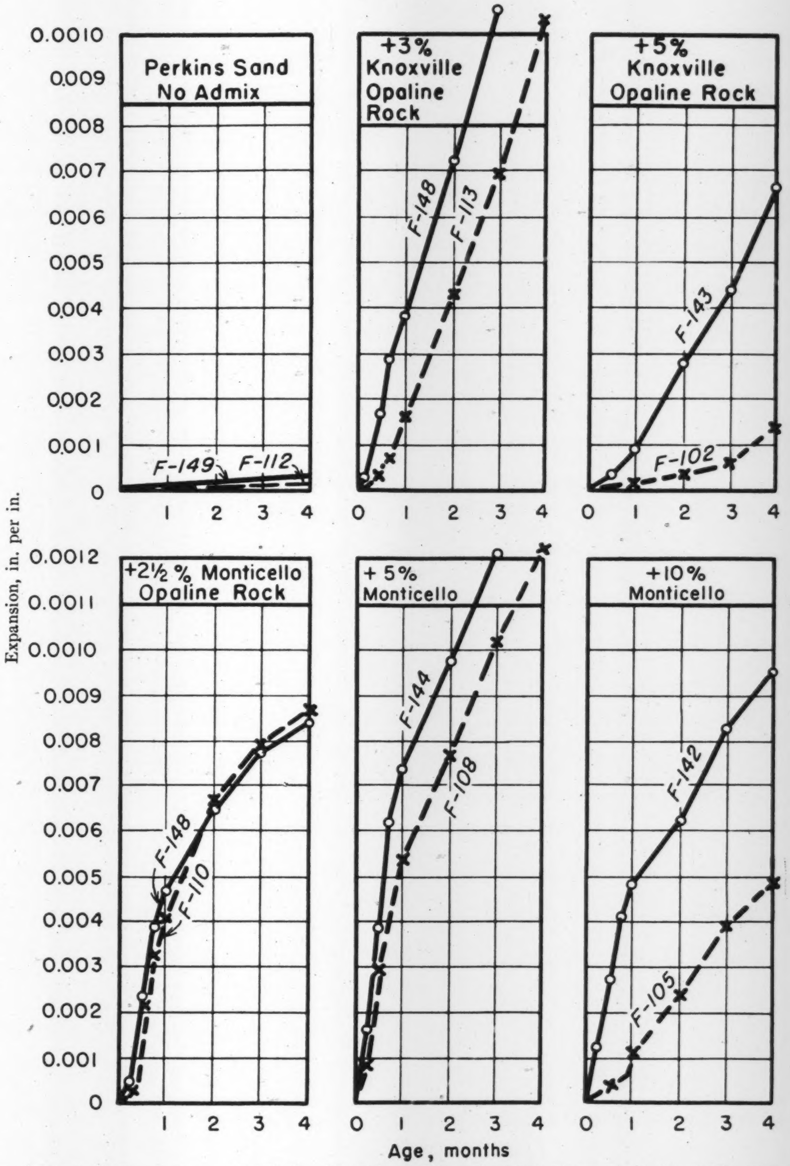


FIG. 9.—Effect of Percentage of Mixing Water on Reactive Admixtures of Variable Percentages of Knoxville and Monticello (Napa Co.). Opaline rocks–Cement G—1.03 per cent Na_2O. Specimen 1 by 1 by 10 in. Mix 1: 2.25.

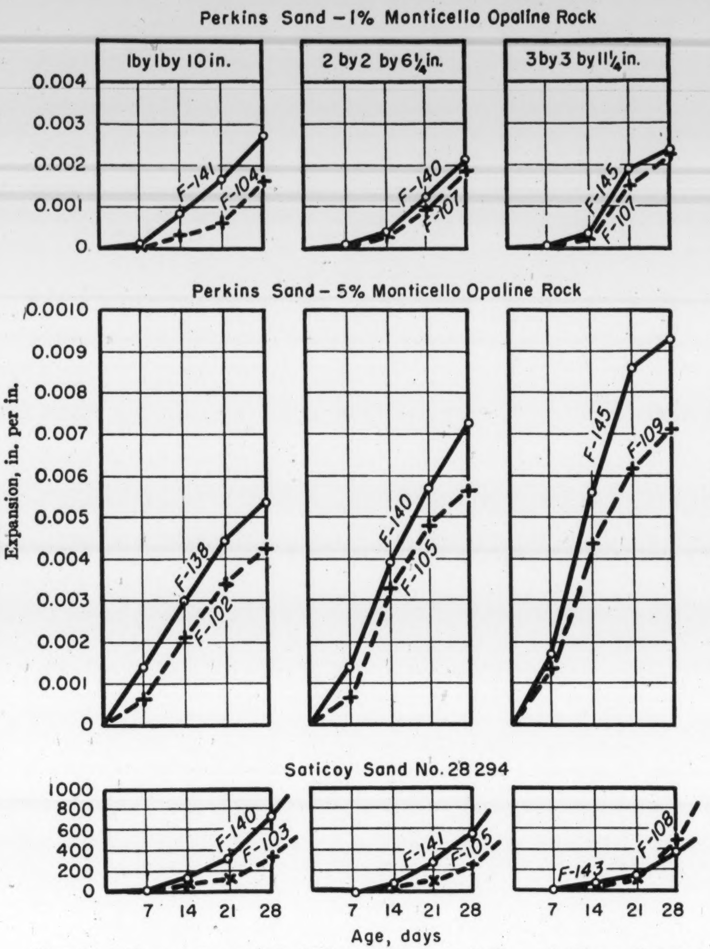


FIG. 10.—Comparison of the Effect of High and Normal Water Cement Ratios onthe Expansion of a Reactive Aggregate High Alkali Cement Combinations of Different Size Test Specimens.

Specimens stored in sealed containers at 100 F. Cement G—1.08 per cent NaO_2 Equivalent. Mix 1:2.25.

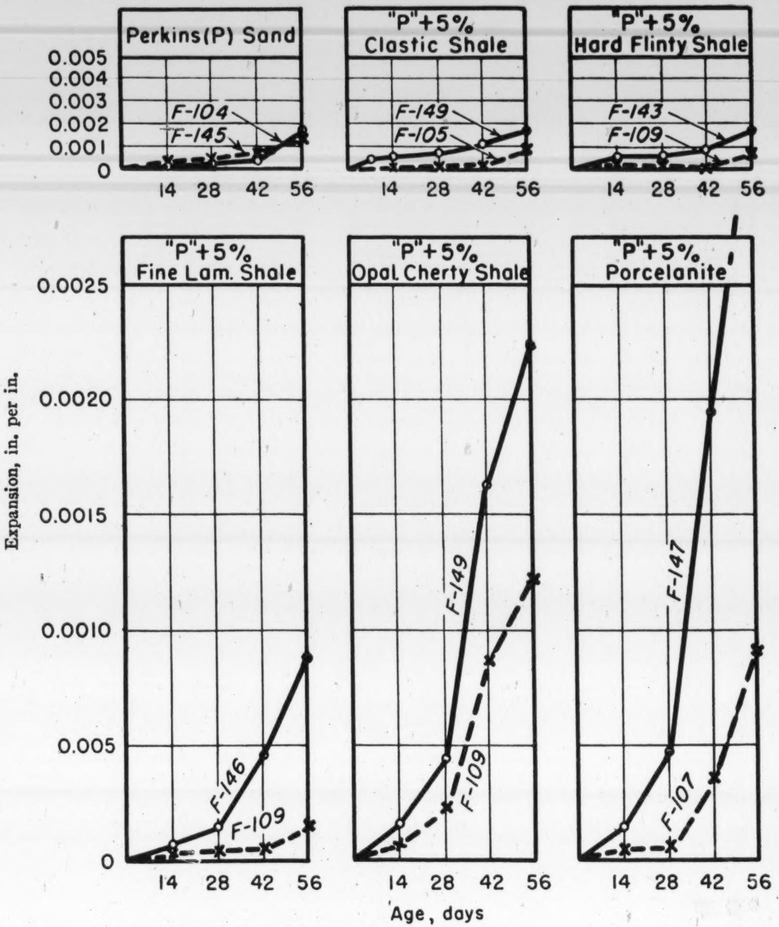


FIG. 11.—Study of the Reactive Properties of the Various Shale Constituents of Sisquoc Sand, Santa Barbara Co.

In addition to determining the reactive characteristics of the different type shales. These tests were designed to determine the accelerating effect, if any, of high water ratios.

1 by 1 by 10-in. Bars stored in sealed containers at 100 F. Cement G—1.08 per cent Na_2O Equivalent. Mix 1:2.25. Fabricated at 73 F, stored at 100 F. High and normal flow.

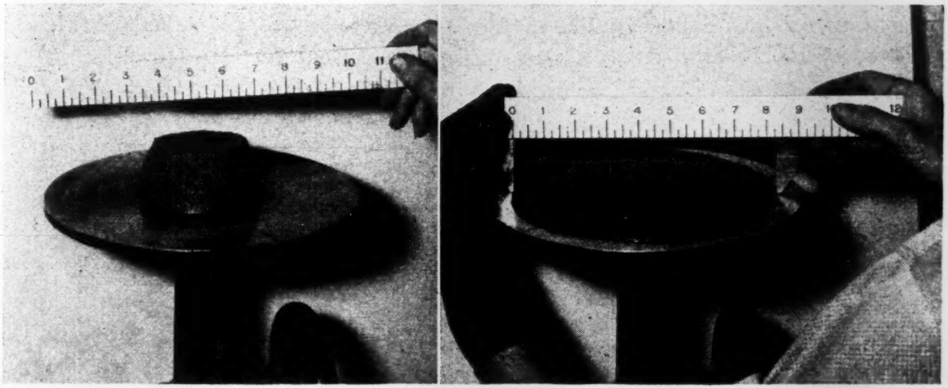
CONCLUSIONS

1. The magnitude of the expansive reaction is dependent on the moisture.

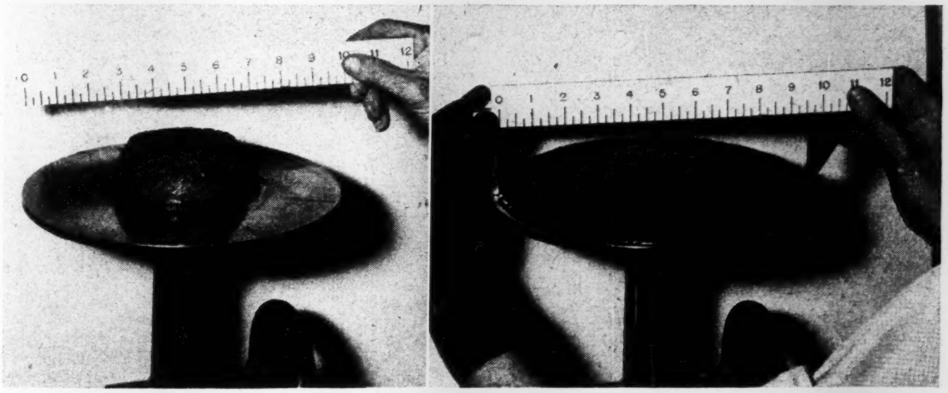
2. Within the limits of the tests conducted, the higher the percentage of mixing water, the greater the expansion.

3. Fabrication and initial curing at low temperatures (40 F) appears to accelerate the expansion with some combinations. It is doubtful however, if the acceleration at 40 F over fabrication and initial 24 hr storage at 73 F is a sufficient on the average, to justify the more difficult procedure.

4. The sensitivity to moisture content and difficulty in establishing optimum moisture for maximum reaction explains in part, at least, the variable performance of different parts of the same structure where the major variables are permeability, per cent of mixing water, and exposure conditions.



Water 13.0 per cent. Flow 107



Water 14.5 per cent. Flow 143.

FIG. 12.—Showing Typical Consistency and Flow of Cement-Aggregate Reactivity Test Mixtures Using Normal and High Water Ratios. 1: 2.25 mix.

Recommendations:

Based on the above observations, it is recommended that further study be given to the effect which an increase in the normal percentage of water may have in accelerating the expansive reaction between the cement and aggregate to the end that if the results of the California tests set forth herein are confirmed by other agencies the procedure currently defined in ASTM Method C 227 be modified to specify that the amount of mixing water shall be sufficient to produce a flow of 140 to 150 instead of 100 to 115.

Acknowledgment:

The tests described and illustrated in this report were conducted by Associate Materials and Research Engineer W. E. Haskell assisted by Assistant Physical Testing Engineer Dan Howe.

FREEZE-THAW RESISTANCE OF CONCRETE AS AFFECTED BY ALKALIES IN CEMENT*

By Bailey Tremper[1]

Synopsis

Concretes made with several aggregates occurring in the State of Washington have exhibited good resistance to freezing and thawing when mixed with low-alkali cements but poor resistance when high-alkali cements were used. These aggregates are "normal" in the properties usually measured and many of them give insignificant expansion when mixed with high-alkali cements and are subjected to the mortar-bar expansion test in sealed containers.

Data of a large number of freezing-thawing tests of concrete containing aggregates from Rock Island, Wash., are presented, together with similar tests using aggregates from Steilacoom, Wash. Resistance to freezing and thawing is shown to be increasingly poor as the alkali content of the cement is increased. Concrete containing Rock Island aggregates is more seriously affected by alkalies than is that containing Steilacoom aggregates.

Rock Island aggregates contain traces of saponifiable organic matter which cause the entrainment of small amounts of air in proportion to the alkali content of the cement. The air thus entrained tends to improve resistance to freezing and thawing but is not formed in sufficient quantity to counteract the inherently poor resistance brought about by the use of high-alkali cement. Additional entrained air introduced by an air-entraining agent improves the resistance to a value that is apparently satisfactory.

In a previous report (**1**)[2] it was shown that concrete containing high-alkali cement, when made with sand and gravel from six sources in Washington, was less resistant to freezing and thawing than concrete having the same aggregates mixed with low-alkali cements. Four of the aggregates were not reactive with alkalies as judged by the mortar-bar expansion test. These results, while applying to aggregates from scattered locations in one state only, may well be applicable to a much larger area.

The purpose of the investigation reported herein was to study more completely the effect of alkali content of the cement on resistance to freezing and thawing of concrete made with one of the non-reactive aggregates used in the previous series. Fifteen cements were included in the present study whereas the original work involved only five and one of these, the highest in alkali content, did not result in the lowest degree of durability. Sand and gravel occurring in a terrace along the Columbia River near the town of Rock Island in central Washington were selected for the present tests because of the pronounced effect of alkalies in the cement on the durability of concrete, as shown in the earlier report, and because of the high proportion of disintegrating concrete found in structures containing aggregates from this source. It was also desired to study the improvement that might result from the use of air entrainment in such concrete.

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* Presented at the Fifty-fourth Annual Meeting of the Society, June 18–22, 1951.

[1] Materials and Research Engineer, Washington Department of Highways, Olympia, Wash.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1107.

SCOPE OF TESTS

The 15 cements used in these tests provided a fairly well graduated series in alkali content ranging from 0.15 to 1.34 per cent Na_2O equivalent. Concrete specimens for freezing and thawing tests were 4 by 4 by 20-in. bars. Each cement was mixed with the Rock Island aggregates. Solutions of sodium hydroxide and potassium hydroxide were added to the mixing water in batches containing the eight cements of lowest alkali content. The solutions were added in amounts equivalent to increasing the alkali content of the cement to 0.50 per cent Na_2O and 0.76 per cent K_2O (1.00 per cent Na_2O equivalent). Vinsol resin solution was added to the mixing water in an amount to produce from 3 to 6 per cent entrained air in the fresh concrete in mixes containing the six cements of highest alkali content, and one low-alkali cement to which additions of NaOH and KOH were made also. Specimens were also made with nine of the cements and non-reactive sand and gravel from Steilacoom, Wash. There was an insufficient supply of the remaining six cements for inclusion in the Steilacoom group. Aggregates from Steilacoom have been used extensively in concrete construction, principally in the Puget Sound area of Washington, and have an excellent service record.

MATERIALS AND TESTS

Aggregates from Rock Island and Steilacoom were obtained from stocks of washed and screened materials from commercial plants at these locations. The composition of the Rock Island gravel by lithological types is as follows:

Granitic	38.2 per cent
Basic extrusive	20.1 per cent
Felsite	11.5 per cent
Gneiss	9.0 per cent
Quartzite	8.7 per cent
Felsite porphyry	6.0 per cent
Acidic intrusive	5.8 per cent
Miscellaneous	0.7 per cent
	100.0 per cent

The gravels were screened and recombined to the desired grading. The Rock Island sand was used in the grading as received. It was quartered while damp to the quantities required for individual batches. Steilacoom sand was screened and recombined to duplicate the grading of the Rock Island sand. The gradings of the aggregates as used are shown in Table I. Additional data on the properties of these aggregates were reported previously (1) and they are considered to be "normal."

TABLE I.—GRADING OF AGGREGATES AS USED IN CONCRETE TEST SPECIMENS.

	Series 1 and 2	Series 3
Fine Aggregate		
Per cent passing U. S. No. 3 Sieve	100	100
No. 4 Sieve	98	98
No. 6 Sieve		92
No. 8 Sieve	84	87
No. 16 Sieve	64	66
No. 30 Sieve	38	39
No. 50 Sieve	10.7	10
No. 100 Sieve	1.3	2
No. 200 Sieve		0.5
Coarse Aggregate		
Per cent passing 1¼-in. Sieve	100	
¾-in. Sieve	50	
½-in. Sieve	24	
¼-in. Sieve	0	

NOTE.—Gradings of Rock Island and Steilacoom aggregates were substantially identical.

Nine of the fifteen cements were from mills in Washington and adjoining states. The remainder were supplied through the courtesy of the U. S. Bureau of Reclamation and the U. S. Bureau of Public Roads. The cements were stored in the laboratory in metal cans with friction plugs. Chemical analyses and physical properties of the cements are given in Table II. The reference number assigned to each cement is the same as the percentage of alkalies (Na_2O equivalent) × 100.

TABLE II.—CHEMICAL ANALYSIS IN PER CENT AND PHYSICAL PROPERTIES OF CEMENTS.

Reference	No. 15	No. 25	No. 51	No. 56	No. 57	No. 63	No. 66	No. 69	No. 79	No. 84	No. 89	No. 96	No. 109	No. 120	No. 134
Na_2O	0.05	0.15	0.05	0.24	0.31	0.17	0.06	0.50	0.05	0.55	0.60	0.68	0.59	0.94	1.25
K_2O	0.16	0.15	0.70	0.48	0.39	0.70	0.91	0.29	1.12	0.44	0.44	0.43	0.60	0.40	0.14
Na_2O equivalent	0.15	0.25	0.51	0.56	0.57	0.63	0.66	0.69	0.79	0.84	0.89	0.96	1.09	1.20	1.34
SiO_2	21.82	22.86	21.41	20.89	21.21	22.00	21.37	21.80	21.70	22.00	22.04	22.04	22.29	24.58	20.89
Al_2O_3	4.83	4.24	4.76	6.03	5.26	4.96	4.82	5.36	5.40	4.92	4.58	5.12	4.71	3.96	5.32
Fe_2O_3	3.68	3.64	3.26	3.27	4.18	3.94	3.63	4.35	2.54	4.02	3.77	4.99	4.01	3.07	4.09
CaO	64.17	64.22	62.44	62.70	63.02	61.94	62.07	63.14	62.17	61.66	61.23	62.91	63.16	62.22	62.84
MgO	1.38	2.19	2.53	2.20	2.61	2.46	2.74	2.17	2.78	3.26	2.72	1.46	2.10	0.86	1.76
SO_3	1.38	1.65	1.75	1.52	1.45	1.81	1.55	1.75	1.86	1.56	1.77	1.69	1.29	1.79	1.73
Loss on ignition	2.70	0.93	3.00	3.20	1.74	2.63	3.26	0.89	2.75	1.80	3.42	1.35	1.36	2.15	2.60
Total	100.37	100.03	99.87	100.53	100.17	100.61	100.41	100.23	100.37	100.21	100.66	100.67	100.11	99.97	100.62
Insoluble residue	0.09	0.50	0.34	0.14	0.20	0.25	0.16	0.27	0.30	0.28	0.11	0.10	0.20	0.63	0.12
C_3S	53	55	50	47	57	41	48	43	43	41	41	42	47	30	51
C_2S	23	14	24	25	18	32	25	31	30	32	32	32	29	48	22
C_3A	7	5	7	10	4	6	7	7	10	7	6	5	6	5	7
C_4AF	11	11	10	10	13	12	11	13	8	12	11	15	12	9	12
Specific surface, sq cm per g	1795		1950	1715	1885	1895	1765	1600	....	1760	1900	2240		1675	1735
Autoclave expansion, per cent	0.03	0.01	0.03	0.05	0.03	0.05	0.07	0.07	....	0.03	0.10	0.02	0.12	0.01	0.02
Time of set, Vicat, initial, hr-min	3–30	2–00	2–45	1–45	3–15	2–30	2–45	2–45	....	2–30	4–15	2–30	2–00	2–30	2–45
Time of set, Vicat, final, hr-min	6–00	5–00	5–30	5–00	6–00	6–00	6–00	6–45	....	6–30	6–30	6–00	5–00	5–30	6–30
Time of set, Gilmore, initial, hr-min	4–30	3–15	3–15	2–45	3–45	3–15	4–00	4–45	....	4–45	4–45	3–45	3–00	3–30	4–00
Time of set, Gilmore, final, hr-min	7–00	6–30	7–00	6–30	7–00	7–00	7–00	8–00	....	8–00	6–30	8–00	6–30	7–00	7–00
Tensile Strength, psi, 3 days	267	286	209	193	242	207	275	268	....	240	225	276	228	206	230
Tensile Strength, psi, 7 days	328	340	289	280	320	300	350	368	....	336	283	340	375	290	312
Tensile Strength, psi, 28 days	400	432	415	385	404	388	410	427	....	428	380	433	439	384	394
Compressive Strength, psi, 3 days	1690	1390	1600	1415	1350	1460	1690	1420	....	1385	1265	1405	985	1120	1185
Compressive Strength, psi, 7 days	2525	1965	2525	2090	2475	2290	2880	2355	....	2325	2080	2325	2065	1940	2100
Compressive Strength, psi, 28 days	3875	3500	4700	4170	3875	4270	4800	4090	....	4575	3610	4415	4240	3600	3340

Concrete for freezing and thawing specimens was mixed in a Lancaster Type SW open pan mixer. Water in excess of that required for saturation was added to each lot of aggregate at least 24 hr before mixing. Water was added during mixing in quantities to result in slumps generally in the range of 3 to 4 in.

The concrete was proportioned to contain 6 sacks of cement per cu yd. Batches were of a size to make four specimens each in Series 1 and two specimens each in Series 2 and 3. Three batches of each cement-aggregate combination were made on different days. In Series 1 and 2, one specimen from each batch was selected for the exposure conditions described below.[3] In series 3, all six specimens were subjected to a single exposure condition. Mixing data are given in Table III.

The percentage of air in the fresh concrete was determined for each batch. In Series 1 and 2, air was determined in accordance with ASTM Method C 138[4] except that the unit weight of the concrete was determined to the nearest gram in a container of 0.1 cu ft capacity. In Series 3, air was determined by the pressure method.

TABLE III.—MIX DATA.

Batch weights of saturated, surface-dry aggregates per sack of cement: Rock Island, fine aggregate, 249 lb; coarse aggregate, 335 lb; Steilacoom, fine aggregate, 249 lb; coarse aggregate, 332 lb. Values shown below are the average of determinations made on each batch.

Cement Reference Number	Alkalies, per cent	Vinsol Resin	Source of Aggregates	Water-Cement ratio, gal per sack	Slump, in.	Air, per cent	Series	Exposure Conditions
15	0.15	No	Rock Island	5.99	3¾	0.89	No. 1	*A*, *B*
25	0.25			5.82	3	1.44	No. 1	
51	0.51			5.86	3¼	0.88	No. 1	
56	0.56			5.72	3¼	1.36	No. 1	
57	0.57			5.65	2¾	1.74	No. 1	
63	0.63			5.68	3¼	1.82	No. 1	
66	0.66			5.63	3½	1.88	No. 1	
69	0.69			5.56	3¼	2.22	No. 1	
79	0.79			5.62	3¼	1.45	No. 1	
84	0.84			5.65	3¾	1.43	No. 1	
89	0.89			5.85	4	1.56	No. 1	
96	0.96			5.56	2½	1.61	No. 1	
109	1.09			5.69	3¼	2.18	No. 1	
120	1.20			5.29	4¼	4.23	No. 1	
134	1.34			5.73	4¼	2.18	No. 1	
25	0.25		Steilacoom	5.62	3¼	1.37	No. 1	
51	0.51			5.52	3½	1.37	No. 1	
56	0.56			5.47	3¾	1.35	No. 1	
57	0.57			5.63	3	1.56	No. 1	
63	0.63			5.68	3¾	0.91	No. 1	
69	0.69			5.55	3½	2.18	No. 1	
84	0.84			5.60	4	0.67	No. 1	
96	0.96			5.58	2¼	1.38	No. 1	
109	1.09			5.44	3½	1.10	No. 1	
15	1.00		Rock Island	5.76	3¾	1.98	No. 2	*B*
25	1.00			5.94	4½	1.97	No. 2	
51	1.00			5.78	3¼	1.57	No. 2	
56	1.00			5.58	3¾	2.19	No. 2	
57	1.00			5.68	3¼	2.20	No. 2	
63	1.00			5.62	2¾	2.34	No. 2	
66	1.00			5.62	4½	1.75	No. 2	
69	1.00			5.70	3	2.98	No. 2	
84	0.84	Yes		5.33	3¾	4.70	No. 2	
89	0.89			5.24	3½	4.45	No. 2	
96	0.96			5.05	2½	4.46	No. 2	
109	1.09			5.32	3¼	4.48	No. 2	
120	1.20			4.91	4¼	5.85	No. 2	
134	1.34			5.25	4¾	5.55	No. 2	
25	0.25	No		5.77	3	1.58	No. 3	*E*
51	0.51			5.47	3¾	1.50	No. 3	
63	0.63			5.20	3¼	1.66	No. 3	
84	0.84			5.32	3½	1.94	No. 3	
96	0.96			5.67	3	2.04	No. 3	
109	1.09			5.35	4	1.86	No. 3	
25	1.00			5.42	3¾	2.91	No. 3	
25	0.25	Yes		4.95	3	3.43	No. 3	
51	0.51			4.85	3½	3.48	No. 3	
63	0.63			4.70	4½	4.96	No. 3	
84	0.84			4.89	4¼	4.62	No. 3	
96	0.96			5.02	4	5.41	No. 3	
109	1.09			4.80	4	5.03	No. 3	
25	1.00			5.63	4	5.07	No. 3	

---

[3] The remaining specimens were exposed to natural weathering and the results are not considered in this paper.

[4] Method of Test for Weight per Cubic Foot, Yield and Air Content (Gravimetric) of Concrete (C 138 - 44), 1949 Book of ASTM Standards, Part 3, p. 872.

Freezing and thawing specimens were cast horizontally in shellacked and greased wood molds. There was some loss of water from the bottom of the molds.

TABLE IV.—RESULTS OF FREEZING-AND-THAWING TESTS.
Durability Factor of 4 by 4 by 20-in. bars after number of cycles shown.

Cement Reference Number	Alkalies, per cent	Vinsol Resin	Source of Aggregates	Air, per cent	Series	Exposure *A*[a]		Exposure *B*	
						78 Cycles	156 Cycles	78 Cycles	156 Cycles
15	0.15	No	Rock Island	0.89	No. 1	97	99	95	93
25	0.25			1.44		97	98	96	95
51	0.51			0.88		88	93	90	79
56	0.56			1.36		95	91	91	91
57	0.57			1.74		95	89	94	94
63	0.63			1.82		97	79	95	95
66	0.66			1.88		95	94	93	93
69	0.69			2.22		96	86	94	95
79	0.79			1.45		94	43	89	81
84	0.84			1.43		93	32	90	78
89	0.89			1.56		95	81	91	90
96	0.96			1.61		95	14	87	85
109	1.09			2.18		94	8	87	81
120	1.20			4.23		92	87	93	93
134	1.34			2.18		94	52	91	86
25	0.25		Steilacoom	1.37		100	110	100	97
51	0.51			1.37		100	109	100	98
56	0.56			1.35		99	78	97	95
57	0.57			1.56		100	110	98	96
63	0.63			0.91		99	92	100	99
69	0.69			2.18		99	106	100	97
84	0.84			0.67		98	76	98	95
96	0.96			1.38		99	60	97	95
109	1.09			1.10		98	75	97	92
15	1.00		Rock Island	1.98	No. 2	....	....	92	70
25	1.00			1.97		....	....	92	80
51	1.00			1.57		....	....	85	52
56	1.00			2.19		....	....	89	86
57	1.00			2.20		....	....	93	91
63	1.00			2.34		....	....	88	91
66	1.00			1.75		....	....	87	67
69	1.00			2.98		....	....	91	89
84	0.84	Yes		4.70		....	....	93	92
89	0.89			4.45		....	....	94	94
96	0.96			4.46		....	....	94	94
109	1.09			4.48		....	....	90	88
120	1.20			5.85		....	....	95	93
134	1.34			5.55		....	....	94	93
25	0.25	No		1.58	No. 3	87	83	....	....
51	0.51			1.50		96	90	....	....
63	0.63			1.66		95	65	....	....
84	0.84			1.94		94	61	....	....
96	0.96			2.04		95	35	....	....
109	1.09			1.86		94	32	....	....
25	1.00			2.91		95	85	....	....
25	0.25	Yes		3.43		98	93	....	....
51	0.51			3.48		95	92	....	....
63	0.63			4.96		96	92	....	....
84	0.84			4.62		96	90	....	....
96	0.96			5.41		97	86	....	....
109	1.09			5.03		96	86	....	....
25	1.00			5.07		95	91	....	....

[a] Exposure *E* in series No. 3.

The method of conducting freezing and thawing tests has been described in detail in a previous paper (2). In brief, the specimens were mounted vertically in metal racks which permitted substantially complete exposure to the air on all sides. The temperature of the bars was lowered to 0 F in about 5 hr after which they were transferred to an adjacent room where they were thawed under sprays of water for 2 hr at which time the temperature at the center of the bars was about 45 F and all accumulated ice on the surface had been melted. Two cycles per day were completed continuously, the bars remaining in the cold room over night, except that at the end of each thirteenth cycle, one cycle was omitted while the dynamic modulus of elasticity was determined.

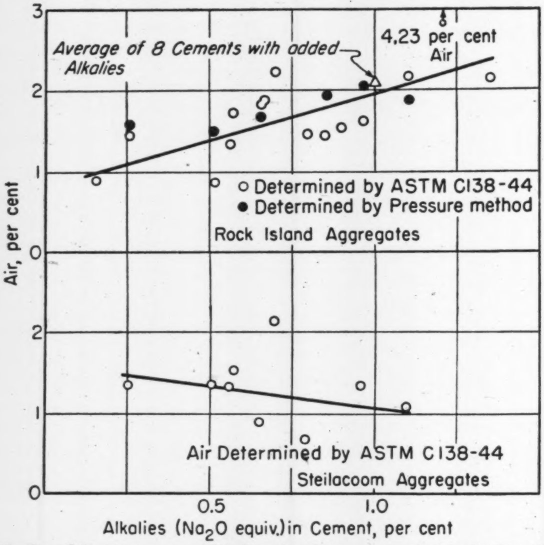

FIG. 1.—Relationship Between Alkalies in Cement and Air in Fresh Concrete.

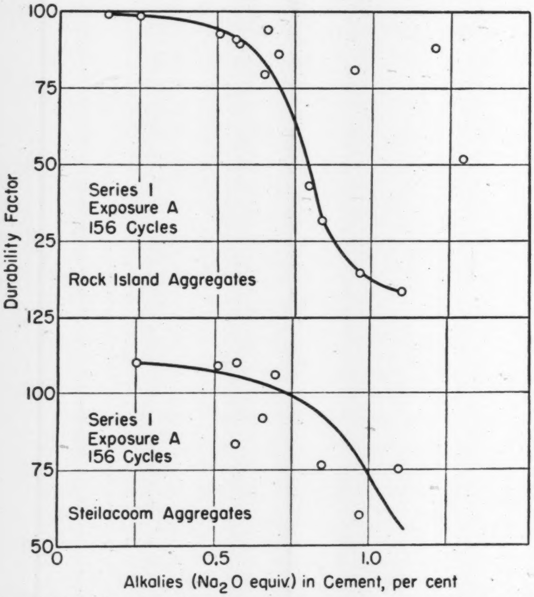

FIG. 2.—Relationship Between Alkalies in Cement and Durability Factor of Concrete.

The effects of freezing and thawing are expressed in this report in terms of "durability factor" as derived by Wuerpel (3). The durability factor expresses the relative loss in dynamic modulus of elasticity, E, at the end of the selected number of cycles. If the loss is less than 50 per cent the factor is simply the loss at the end of the last cycle. For specimens that suffer more than 50 per cent loss, the durability factor is computed by formula from the number of cycles at which 50 per cent loss was reached. This formula is useful because it is difficult to measure low values of dynamic E accurately.

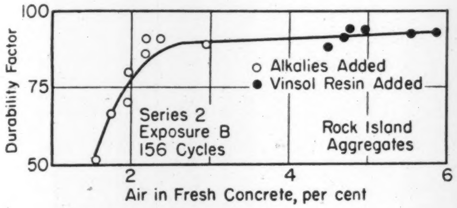

FIG. 3.— Effect of Entrained Air on Durability, Exposure B.

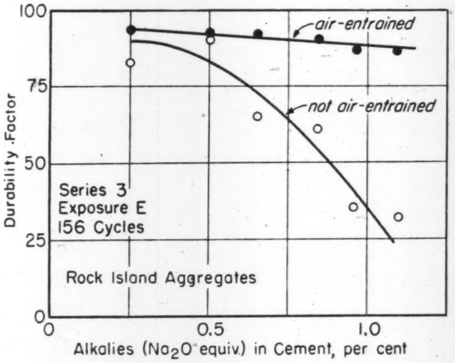

FIG. 4.—Effect of Entrained Air on Durability, Exposure E.

The sequence of molding and the exposure conditions to which the bars were subjected are as follows:

Series 1—Exposure Conditions A and B:

288 bars representing 15 cement combinations with Rock Island aggregates and 9 cement combinations with Steilacoom aggregates. Dates of Mixing: October 23 to November 8, 1944 (concurrent with Series 2).

Series 2—Exposure Condition B:

84 bars representing 14 cement combinations with Rock Island aggregates. Alkalies in the form of NaOH and KOH were added to the mixing water with the eight cements of lowest alkali content. Vinsol resin solution was added to the mixing water with the six cements of highest alkali content. Dates of mixing: October 23 to November 8, 1944 (concurrent with Series 1).

Series 3—Exposure Condition E:

86 bars representing 14 cement-admixture combinations with Rock Island aggregates. Cements 25, 51, 63, 84, 96 and 109 were used plain and with Vinsol resin. In addition, alkalies were added to cement 25 both with and without Vinsol resin. Dates of mixing: January 27 to January 30, 1947.

Exposure Condition A:

1. 1 day in molds.
2. 27 days in fog room at 70 F.
3. 78 cycles of freezing and thawing.
4. 8 months in fog room at 70 F.
5. 78 cycles of freezing and thawing.

Exposure Condition B:

1. 1 day in molds.
2. 6 months in fog room at 70 F.
3. 78 cycles of freezing and thawing.
4. 8 months in fog room at 70 F.
5. 78 cycles of freezing and thawing.

Exposure Condition E:

This duplicates exposure condition A in all respects except that the initial fog room storage, item 2, was for 20 days.

DATA OF THE TESTS

Table IV gives the results of freezing and thawing tests.

Figure 1 shows the relationship between alkali content of the cement and the percentage of air in the fresh concrete.

Figures 2 to 4 inclusive show the relationship between alkali content of the cement and durability factor after freezing and thawing and also the effect of air entrainment.

DISCUSSION OF TEST RESULTS

Figure 1 indicates a definite trend toward increased air in the fresh concrete made with Rock Island aggregates as the percentage of alkalies in the cement increases. The plotted curve indicates an increase of about 1.4 per cent air for the cement of highest alkali content over that of the lowest alkali content. Cement 120 has air-entraining properties and it entrained air considerably in excess of that indicated by the plotted curve. Cement 69 also appears to have mild air-training properties. Low-alkali cements that were adjusted to an alkali content of 1.00 per cent by the addition of NaOH and KOH, entrained air to an extent that agreed quite well with the plotted curve.

Figure 1 also shows corresponding air contents for those cements that were mixed with Steilacoom aggregates. The trend suggests slightly decreasing amounts of air as the alkali content of the cement increases. With this combination, as with Rock Island aggregates, cement 69 was found to entrain an additional amount of air. Cement 120 was not used in the Steilacoom group.

It is indicated, therefore, that the effect of increasing the air content of concrete is a property of Rock Island aggregates not common to Steilacoom or, presumably, other aggregates. It was noted during the molding of Rock Island specimens that part of the air bubbles formed by the high-alkali cements seemed to be exceptionally large and that some escaped from the concrete during the process of puddling and striking off. Loss of air during molding undoubtedly was responsible for minor inconsistencies in the determined values of air content. Another source of discrepancy probably was a variance in the percentage of total alkalies in the cement that went into solution during the mixing period.

The constituent in the Rock Island aggregates that is responsible for air entrainment appears to be a minute quantity of saponifiable organic matter. This is indicated by the following tests:

1. When Rock Island sand was shaken with water, any bubbles formed disappeared within 10 sec.
2. When shaken with two per cent NaOH solution, a foam was produced that persisted for 60 sec.
3. When the sand mixed with cement was shaken with two per cent NaOH solution, a foam was formed that persisted for several hours.
4. Extraction of Rock Island sand with ether yielded 0.01 per cent of a brown, viscous to semi-solid, oily material that was slightly or incompletely soluble in water but which was readily soluble in dilute sodium hydroxide solution.
5. The sand after extraction with ether, when subjected to treatment 3, produced a minor amount of foam of weak persistence.
6. Steilacoom sand when subjected to treatment 3 yielded no foam of measurable persistence.
7. No ether-soluble material was found in Steilacoom sand.

The presence of a saponifiable organic compound in the Rock Island aggregates may be explained by a theory that this locality was once a lake caused by the damming of the lower Columbia River in the geologic past. Residues from organisms living in the lake could be the origin of saponifiable matter now found in the Rock Island deposit. A condition similar to this now exists in Soap Lake, a body of water about 30 miles east of Rock Island.

The saline water of the lake is reported to contain a measurable amount of water-soluble oil thought to be the remains of living creatures or of anaerobic bacteria that are present in the water. If oil was formed at Rock Island under similar conditions, that which remained in the sand and gravel deposit resists solution by fresh water too strongly to be removed by washing in the preparation of concrete aggregates.

Tests of a number of other sands in Washington indicate that the presence of ether-soluble material is not common;

however, it has been found in sands from the Blue River and Republican River in Kansas.

Figure 2 shows the variation in durability factor of Rock Island concrete with alkali content of the cement resulting from exposure condition *A* of Series 1. Greatly reduced resistance to freezing and thawing is indicated for the cements of higher alkali content. The curve has been drawn in an "S" shape because it seems logical to do so. It would not be unexpected to find little change in resistance among cements in the low range of alkali contents. In the lower range of durability, values cannot be less than zero and, therefore, a flattening of the curve is indicated. There are three cements, 89, 120 and 134, that do not agree with the curve as drawn. The results for cement 120 can be explained by its air-entraining properties. No explanation is offered for the behavior of cements 89 and 134.

Figure 2 also shows the variation in durability factor of Steilacoom concrete with alkali content of the cement resulting from exposure *A* of Series 1. It will be noted that although the cements of higher alkali content had lower resistance, the effect is much less pronounced than with Rock Island concrete.

Table III shows that none of the cements was greatly affected by the first 78 cycles of freezing and thawing. The intervening period of storage in the fog room evidently was necessary to develop the lack of resistance that was observed during the second exposure to freezing and thawing. The changes that occurred in the specimens during fog room storage are not known. It is evident, however, that prolonged fog room storage ahead of freezing and thawing does not result in lack of resistance. This is demonstrated by exposure condition *B* of Series 1. It will be noted from Table III that the durability factors after the first 78 cycles of exposure *B* are not significantly lower than under exposure *A* after the same number of cycles. The durability of both Steilacoom and Rock Island concrete was greatly improved by the longer preliminary moist curing.

In Series 2 the specimens were subjected to exposure *B*. The alkali content of the eight cements having the lowest percentage of alkalies was adjusted to the equivalent of 1.00 per cent Na_2O by additions of NaOH and KOH. Thus in the entire series, all cements may be considered to contain at least 0.84 per cent Na_2O equivalent. In Fig. 3, the durability factor of these cements is plotted against air content. The curve indicates increasingly better resistance as the air content approaches a value of 3 per cent. The air formed by interaction of alkalies in the cement and the saponifiable matter in the aggregates seems to be effective in increasing durability provided sufficient air is thus entrained. The amount of air entrained by this means, however, is uncertain and seems to depend on circumstances connected with the mixing and placing procedure that cannot be controlled dependably. When an air-entraining agent is used to make certain that the air content is above 3 per cent, the durability factor is uniformly high. Figure 3 seems to demonstrate that the air entrained by alkalies in the cement is not itself responsible for the poor durability exhibited by the Rock Island aggregates when mixed with high-alkali cements. It seems unlikely that the saponifiable matter itself is responsible for poor durability because, in that case, it should be equally deleterious with low-alkali cements.

The long (6-month) preliminary curing used in exposure *B* to which the specimens of Series 2 were subjected has been shown to increase the durability above that that might be expected by a more practical, shorter period of initial curing. Series 3 was started at a later date to determine if air-entrainment could be depended upon to result in good durability when the initial curing period was relatively short. It was necessary to omit a number of cements in this series because the supply of some had been exhausted. The sequence of curing, freezing and thawing, and intervening fog room storage followed closely that of exposure *A* of Series 1. Results of Series 3 are plotted in Fig. 4. It will be noted that the data for non-air-entrained concrete agree quite well with those of Series 1. The addition of an air-entraining agent in amounts to bring the total air content of the fresh concrete to values between 3 and 6 per cent was found to be effective in increasing the resistance to freezing and thawing to the point that high-alkali cements gave substantially as good durability as the low-alkali cements. The laboratory tests thus indicate that Rock Island aggregates may be used satisfactorily with high-alkali cements if air-entraining agents are used in the proper amount.

It will be noted in Table IV that when the alkali content of cement 25 was increased to 1.00 per cent Na_2O equivalent, the durability factor of the non-air-entrained concrete was relatively high for this alkali content. In the light of the preceding discussion, this result may be explained by the somewhat fortuitous entrainment of 2.91 per cent air in this concrete.

As mentioned above the test data do not provide an adequate explanation of why high percentages of alkalies in the cement lower the durability of concrete containing Rock Island aggregates and, to a less degree, of concrete containing Steilacoom aggregates. Neither is there a sufficient explanation of why the poor durability results only when the specimens are subjected to an intervening period of moist storage during the freezing and thawing tests. It is possible that these results could have been explained in terms of freezable water in the concrete according to Powers and Brownyard (**4**) and of air-void space as developed by Sweet (**5**). These studies were not published at the time these tests were started and the data obtained are not sufficient to permit calculations of these factors.

DISCUSSION OF FIELD EXPERIENCE

An alarming degree of disintegration has been observed in the superstructures of concrete bridges made with Rock Island aggregates and a cement containing about 0.85 per cent alkalies. After these results became known it was the practice of the Washington Department of Highways for several years to limit the percentage of alkalies in portland cement used in Rock Island aggregates to a maximum of 0.60 per cent. Later, acting upon the favorable results indicated by the laboratory tests, the restriction on alkali content of the cement was removed but the concrete was required to be air-entrained. In August and September, 1946 air-entrained concrete made with Rock Island aggregates and portland cement containing 0.80 per cent alkalies (Na_2O equivalent) was used in the construction of combination sidewalks and ramp driveways. The concrete is subjected to a large amount of vehicular cross travel and de-icing salts are applied frequently during the winter. The concrete has been examined recently and, except for a short section that has ravelled slightly, there are no signs of distress.

The Rock Island pit has since been abandoned as a commercial operation and good opportunities for judging the effectiveness in the field of air-entrainment in concrete made with these aggregates and high-alkali cement are limited to the work mentioned above. Although the length of exposure, 4 yr, is rather short, the results to date are encouraging, particularly so when signs of distress in non-durable concrete have been noted elsewhere as early as 2 yr after construction.

CONCLUSIONS

Aggregates from the Rock Island pit produce concrete that is progressively poorer in resistance to freezing and thawing as the percentage of alkalies in the cement increases. Longer periods of curing before exposure to freezing and thawing conditions, reduce the degree of deterioration but the required length of the curing period is greater than is ordinarily obtainable in construction.

During the mixing of concrete containing Rock Island aggregates the quantity of air entrained increases progressively as the percentage of alkalies in the cement increases. With cements of extremely high alkali content, the increase in entrained air may amount to as much as 1.4 per cent over that entrained by low-alkali cement. The air thus entrained appears to be the result of interaction between the cement and a very small amount of saponifiable organic matter that is present in the aggregates. This excess air, however, is not dependably sufficient in amount to overcome the inherently poor resistance to freezing and thawing exhibited by Rock Island - high-alkali cement combinations. The poor durability of high-alkali cement concrete seems to result from a factor that is not connected with the trace of saponifiable matter that is present in the aggregates.

The entrainment of from 3 to 6 per cent total air by the use of suitable air-entraining agents improves the resistance of high-alkali cement concrete containing Rock Island aggregates to the point where it appears to be equal in resistance to concrete containing low-alkali cement.

The adverse effect of high-alkali content in cement on resistance to freezing and thawing has been noted in concrete made with other aggregates. Although not confirmed by test, it seems probable that such concrete would also be improved by air entrainment.

REFERENCES

(1) Bailey Tremper, "The Effect of Alkalies in Portland Cement on the Durability of Concrete", *Journal*, Am. Concrete Inst., November 1944; *Proceedings*, Vol. 41, p. 89 (1945).

(2) Bailey Tremper, "Symposium on Freezing-Thawing Tests," *Proceedings*, Am. Soc. Testing Mats., Vol. 46, p. 1227 (1946).

(3) Charles E. Wuerpel, discussion of paper by Stanton Walker, "Freezing and Thawing Tests of Concrete made with Different Aggregates," *Journal*, Am. Concrete Inst., Supplement, November 1944; *Proceedings*, Vol. 40, pp. 580–581 (1944).

(4) T. C. Powers and T. H. Brownyard, "Studies of the Physical Properties of Portland Cement Paste, Part 8," *Journal*, Am. Concrete Inst., April, 1947; *Proceedings*, Vol. 43, p. 933 (1947).

(5) Harold S. Sweet, discussion of paper by Jack B. Blackburn, "Freeze and Thaw Durability of Air-entrained Concrete using Indiana Aggregates," *Proceedings*, Highway Research Board, Vol. 28, p. 187 (1948).

AGGREGATES TESTED BY ACCELERATED FREEZING AND THAWING OF CONCRETE*

By W. H. Price[1] and D. G. Kretsinger[1]

Synopsis

The automatic freezing-and-thawing apparatus used for testing concrete in the laboratory of the Bureau of Reclamation at Denver is described and the results are compared with those obtained with the manually operated apparatus used prior to 1946. The procedure employed in determining the suitability of aggregates by testing them in concrete subjected to alternate cycles of freezing and thawing is outlined, and the results obtained with aggregates from a number of projects are presented. The effect of variation in the test procedure is discussed.

The automatic freezing-and-thawing apparatus was installed in the Denver laboratory of the Bureau of Reclamation in 1946. Prior to that time the behavior of concrete had been studied for many years in the smaller manually operated freezing-and-thawing apparatus described before the Society in 1946 **(1)**.[2] It is possible to obtain 50 alternate cycles of freezing and thawing a week and test 800 3 by 6-in. specimens at one time in the new apparatus. The capacity of the equipment was increased sufficiently by the installation of the automatic unit to permit the testing of all aggregates submitted for use in Bureau structures by subjecting concrete containing the aggregate to the freezing-and-thawing test.

In the procedure used in testing the aggregates, which will be described in detail later, duplicate sets of specimens containing the aggregate are made with concrete containing about 5 per cent entrained air and with no purposely entrained air in the concrete. In many cases the tests show that a concrete of satisfactory durability cannot be produced with a given aggregate unless the concrete contains entrained air. Other results, such as those obtained from concretes made with aggregates from the Republican River in Kansas, show that a concrete of good quality cannot be made with this aggregate even with entrained air. In the case of the Republican River aggregates, the test results are in agreement with the field behavior of the concrete.

The durability results are influenced by many variables, some of which will be discussed, and when these variables are properly evaluated this test is considered one of the most reliable means of determining the potential weathering resistance of concrete in a comparatively short time.

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* Presented at the Fifty-fourth Annual Meeting of the Society, June 18–22, 1951.

[1] Head, Materials Laboratory and Materials Engineer respectively, Bureau of Reclamation, Denver, Colo.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1117.

The Automatic Unit

In 1946 the manually operated freezing-and-thawing apparatus described in the earlier paper (1) was replaced by the fully automatic apparatus shown in Fig. 1. In this new apparatus the specimens are frozen and thawed in water as was done previously, but they are handled only once each week when they are measured to determine the effect of the freezing-and-thawing cycles on the concrete.

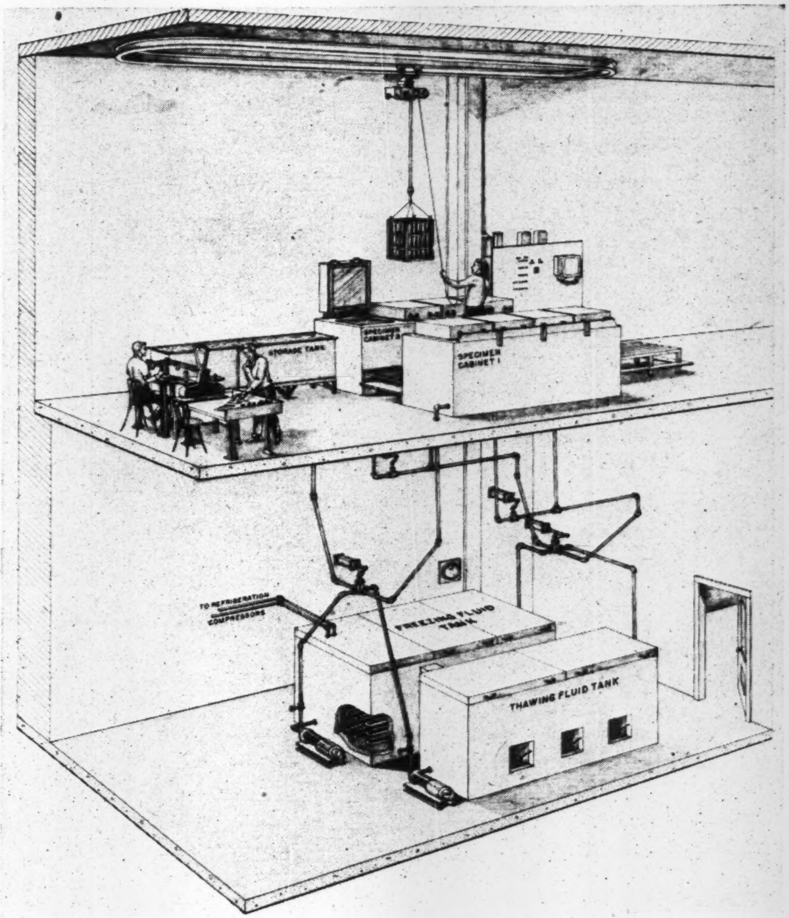

STORAGE TANK
SPECIMEN CABINET 2
SPECIMEN CABINET 1
TO REFRIGERATION COMPRESSORS
FREEZING FLUID TANK
THAWING FLUID TANK

Fig. 1.—Automatic Freezing-and-Thawing Equipment.

The automatic unit consists of two specimen cabinets, a freezing fluid tank, a thawing fluid tank, means for refrigerating and warming the fluids in these tanks, and the pumps, valves, and automatic electrical controls necessary to circulate the fluid through the system. Originally a 60 per cent solution of grain alcohol and water was used as the freezing-and-thawing fluid in the automatic unit, but because of high evaporation losses, severe corrosion of the tanks, and fire hazard, it was replaced by a 25 per cent solution of calcium chloride brine after a few months of operation. Small amounts of sodium hydroxide and sodium dichromate are used to inhibit corrosion by the brine.

This apparatus has been built on two levels; the specimen cabinets are on the mezzanine and the freezing-and-thawing fluid tanks are on the lower floor. Each specimen cabinet can accommodate 400 3 by 6-in. cylindrical specimens. Refrigerated brine is pumped up into one specimen cabinet and returned to the freezing fluid tank by gravity through an overflow weir in the cabinet. Simultaneously, brine which is heated by electric resistance heaters is circulated in a similar manner through the other specimen cabinet. After 1 hr and 25 min, the pumps are stopped, the fluids are drained from the specimen cabinets back into their respective tanks, fluid paths are interchanged and the pumps are restarted. Thus the previously frozen specimens are thawed while the previously thawed specimens are frozen. The fluid paths are controlled by two four-way and two two-way plug type valves which are operated by double acting air cylinders which are activated by solenoid air valves. The drains in the bottoms of the specimen cabinets, controlled by the two-way air-operated plug type valves, are closed during the operation of the cycle and are open for 5 min at the end of each cycle to permit drainage. Fully automatic operation is effected by a clock-driven cam which actuates microswitches which, in turn, operate the pumps and valves through a system of relays and solenoid air valves. An electrically operated cycle counter is used to stop the apparatus after a predetermined number of cycles. Overflow protection is obtained in the specimen cabinets by electrodes placed slightly above the normal brine level. Brine temperatures are thermostatically controlled, and a continuous record of both brine and specimen temperatures is obtained.

Refrigeration is supplied by ammonia compressors which also supply refrigeration for other curing and freezing rooms of the laboratory. Since January, 1950, a freezing brine temperature of 8F and a warm brine temperature of 73 F have been maintained in the automatic freezing-and-thawing apparatus. These brine temperatures produce temperatures at the center of the 3 by 6-in. concrete specimen in the 1 hr and 25 min of freezing and thawing of 10 F and 70 F, respectively. Fifty cycles of freezing and thawing with time allowed for measurement are obtained each week.

TABLE I.—GRADING OF FINE AND COARSE AGGREGATE USED FOR AGGREGATE ACCEPTANCE TEST.

Fine Aggregate Screen Size		Coarse Aggregate Screen Size	
No. 8 to No. 4	15 per cent	⅜ to ¾ in.	60 per cent
No. 16 to No. 8	15 per cent	No. 4 to ⅜ in.	40 per cent
No. 30 to No. 16	25 per cent		
No. 50 to No. 30	24 per cent		
No. 100 to No. 50	16 per cent		
Pan	5 per cent		

AGGREGATE ACCEPTANCE TEST

Samples of aggregate tested in the freezing-and-thawing tests are separated into their component sizes and the fractions smaller than $\frac{3}{4}$-in. in size are recombined for the concrete mix in the proportions shown in Table I.

Recent specifications of the Bureau of Reclamation require that all concrete contain purposely entrained air. Because entrained air improves the resistance of the concrete to freezing and thawing, it is expedient that the suitability of aggregate for concrete be re-evaluated in the light of this improvement. Two mixes are usually made for each aggregate, one with an air-entraining agent and the other without an agent. These concretes are designed for a net water-cement ratio of 0.51 and a slump between 2.5 and 3.5 in. Since the aggregate is used in a dry condition it is necessary to correct for the water absorbed by the aggregate in computing the net water-cement ratio. The 30-min absorption of the aggregate is used as the basis of this adjustment. The plain concrete contains 50 per cent sand, and the entrained air concrete contains 45 per cent sand by weight of the total aggregate. A 5 per cent solution of Vinsol resin is used as the air-entraining agent, and the air content of the concrete is controlled between 4.5 and 5.5 per cent. A blend of several Type II cements from various mills in the Rocky Mountain region is used for these tests.

The aggregate and cement is dry mixed in a pan type mixer for 30 sec before the water is added. After the addition of the water, the concrete is mixed for 3 min. In the case of entrained air concrete, the Vinsol resin solution is added to the mix water, replacing a volume of water equal to the volume of the solution. At the end of the mixing period, standard slump and unit weight measurements are made. Two air content determinations are made on each mix, one with a Washington type air meter and the other gravimetrically. Immediately after these measurements are completed, two 3 by 6-in. cylinders with inserts in each end are made from an undisturbed portion of each mix for freezing-and-thawing tests, and two specimens are made without inserts for compressive strength tests. The cylinder molds are filled in two equal layers; each layer is vibrated internally with a $\frac{3}{8}$-in. diameter rod attached to the end of a small laboratory type internal vibrator. All specimens are cured in their molds in the fog room at 73 F and 100 per cent relative humidity for 24 hr. At the end of 24 hr, the cylinders are removed from their molds and returned to the fog room for an additional 27 days.

The 3 by 6-in. freezing-and-thawing cylinders have conical indentations cast into their ends in which corrosion-resistant metallic inserts are embedded. These gage points are used for length change measurements and are placed in the conical indentations to prevent injury to them when the specimens are placed end to end in the rubber containers. At the end of the 28-day fog curing period, the specimens are put into the rubber containers for protection from the brine, water is added to the containers to a point just slightly above the top specimens, and they are then subjected to alternate cycles of freezing at 10 F and thawing at 70 F. Fifty such cycles are obtained each week. Once each week the specimens are measured for length change and weight loss. Tests are repeated until the specimens have lost 25 per cent of their weight, this being considered as failure. The method of measuring and weighing the specimens and the type of rubber containers used in the test are described in the earlier paper (1).

Comparison of Manual and Automatic Units

Efficient operation of the automatic unit does not permit complete duplication of the procedure used with the manually operated unit. Under the manual procedure, the specimens remained in the thawing tank overnight and weekends, and only ten cycles of freezing and thawing were obtained each week as compared to the 50 cycles possible with continuous operation of the automatic unit.

In the manual unit, the temperature of the freezing brine averaged 5 F, and the center of the specimen reached a temperature below 15 F in 1 hr and 30 min. For a considerable period after starting the operation of the automatic unit it was found difficult to maintain a brine temperature of 5 F, but since January 1950 when new compressors were installed, some of the valves eliminated, the length of pipe lines reduced, and electrical controls simplified and improved, it has been possible to obtain and maintain low brine temperatures. When the automatic unit was operated with a freezing brine temperature of 5 F, the centers of the specimens reached a temperature of 8 F in 1 hr and 25 min, indicating that there was a better circulation of brine in the new unit. During these tests, it was shown that the automatic unit must be operated at a freezing brine temperature of 3 F with temperatures at the center of the specimens of 5 F to approach the same number of cycles required to produce failure in concrete of a given quality in the manual unit. Because of the desire to obtain a greater spread in the number of cycles necessary to produce failure of weaker concrete in the automatic unit, the freezing brine temperature was raised to 8 F. This temperature produces a temperature of 10 F at the center of the specimens in 1 hr and 25 min. Table II is a comparison of the number of cycles required to produce failure of concretes of various qualities in the manual and automatic units for temperatures of 10 F at the center of the specimens.

TABLE II—CYCLES OF FREEZING AND THAWING TO 25 PER CENT WEIGHT LOSS.

Rating of Quality of Concrete	Manual	Automatic
	10 Cycles per Week at 10 F and 70 F	50 Cycles per Week at 10 F and 70 F
Poor	Less than 75	Less than 150
Fair	75 to 150	150 to 300
Satisfactory	...	300 to 500
Good	150 to 300	500 to 1000
Excellent	Over 300	Over 1000

Table II shows that at least twice as many cycles of freezing and thawing are required for failure with concrete of a given quality in the automatic unit than in the manual unit, even though the number of cycles obtained per week were materially greater in the automatic unit.

CONTROL SPECIMENS

Control specimens are made with local aggregate and the standard laboratory cement with every group of project aggregates tested so that variations in the results due to possible changes in the apparatus or test procedure might be ascertained. Since 1950 quite uniform results have been obtained with these control specimens. The number of cycles required for failure of plain concrete has varied from 180 to 250 with a coefficient of variation of 13.4 per cent, while the cycles for the air-entrained concrete has varied from 880 to 1140 with a coefficient of variation of 3.7 per cent. These values were obtained from six plain and six entrained air control mixes made and tested over a period of one year.

COMPARISON OF AGGREGATES

Table III compares the results obtained on some of the aggregates tested in the automatic freezing-and-thawing tests. It will be noted that plain concrete containing Grand Coulee aggregate withstood 600 cycles and air-entrained concrete with this aggregate withstood 1980 cycles. Concrete made with this aggregate has performed very satisfactorily in the field. Aggregate from the Republican River, by comparison, withstood only 90 cycles for the plain concrete and 210 for the air-entrained concrete. The service record of this aggregate is poor. It is difficult to correlate laboratory tests with the service behavior of structures in the field, but a study of all results shows that, where accelerated laboratory tests are properly made and evaluated, reliable indications of the potential weathering resistance of the concrete are possible.

EXPANSION OF CONCRETE

The expansion of the concrete at time of failure is also included in Table III. It should be noted that those concretes which failed early had comparatively high expansion at time of failure. It is sometimes desirable that an indication of the total cycles required to fail the concrete be obtained in advance of actual failure. From the rate of expansion and number of cycles required to produce 0.5 per cent expansion, it is possible to predict the total cycles required for failure. Usually plain concrete will fail within 100 to 150 cycles after reaching 0.5 per cent expansion, and entrained air concrete will fail within 300 to 500 cycles after this expansion is reached.

TABLE III.—DURABILITY RESULTS OF CONCRETES CONTAINING VARIOUS PROJECT AGGREGATES.

Structure	Source and Type of Aggregate Used in Test Specimens	Field Service Record[a]	Plain Concrete		Entrained-Air Concrete	
			Cycles at 25 Per Cent Weight Loss	Per Cent Expansion at 25 Per Cent Weight Loss	Cycles at 25 Per Cent Weight Loss	Per Cent Expansion at 25 Per Cent Weight Loss
Grand Coulee Dam	Natural aggregate deposit from near dam site	Excellent	600	0.76	1,980	0.15
Laboratory aggregate	Natural aggregate deposit near Denver, Colorado	Satisfactory	180	1.48	970	0.60
Kansas-Nebraska investigation	Natural aggregate deposit from Republican River near Indianola, Nebraska[c]	Poor	90	1.54	210[b]	1.10
Kansas-Nebraska investigation	Natural aggregate deposit from Lodgepole Creek near Kimball, Nebraska	Poor	30	...	290[b]	0.86
Kansas-Nebraska investigation	Limestone from quarry near Moline, Kansas	Poor	130	1.00	480	1.46
Hungry Horse Dam	Natural aggregate deposit near dam site	...	350	0.88	1,200	0.28
Hungry Horse Dam	Natural sand and gravel from pit near dam site		200	0.50	750	0.24
Green Mountain Dam	Natural aggregate deposit near Kremmling, Colorado	Fair	150[b]	0.30	...	...
Shasta Dam	Natural aggregate deposit near dam site	...	430[b]	0.30	...	...
Buffalo Rapids Dam	Natural aggregate deposit near dam site	Satisfactory	390[b]	0.18	1,040[b]	0.16
Grassy Lake Dam	Natural aggregate deposit near dam site	Fair	330[b]	0.40	640[b]	0.20
Vallecito Dam	Natural aggregate deposit near dam site	Poor	160	1.40	...	...
Gibson Dam	Natural aggregate deposit near Fairfield, Montana	Very good	440	0.17	...	...
Salt Lake Aqueduct	Natural aggregate deposit near American Fork, Utah	Fair	130	0.90	...	...
Moon Lake Dam	Natural aggregate deposit near dam site	Very good	350	0.34	...	...
Anchor Dam	Natural aggregate deposit near dam site	...	...	...	1,170	0.42
Anchor Dam	Natural aggregate deposit on creek near dam site	...	210	0.76	800	0.40
Angostura Dam	Natural fine and crushed limestone coarse	...	480	1.08	730	0.17
Angostura Dam	All crushed limestone	...	380	0.60	...	...
Friant Dam	Natural aggregate deposit near dam site	...	210	0.30	...	...
Yellowtail Dam	Natural aggregate deposit near dam site	...	170	0.96	700	0.40
Canyon Ferry Dam	Natural aggregate downstream from the dam site	...	170	0.96	1,400	0.30
Cedar Bluff Dam	Natural fine and crushed limestone coarse aggregate	...	70	1.10	600	1.20
Cambridge Diversion Dam	Natural fine and crushed dolomite coarse aggregate	...	100	1.41	550	0.90
Cambridge Diversion Dam	All crushed dolomite	...	80	1.10	880	0.74

[a] Based on plain concrete.

[b] Durability values corrected to 0.51 water-cement ratio. All other concretes made with 0.51 water-cement ratio.

[c] ⅜-in. max. size "sand-gravel" concrete, all other concretes contain ¾-in. max. size aggregate. Entrained air "sand-gravel" concrete contained 8 per cent air and all other concretes contained 5 per cent air.

NOTE. Cement contents for plain concretes ranged from 5¾ to 7¼ sacks per yd and from 4¾ to 6½ for entrained-air concretes, the averages being 6¾ and 6 sacks per yd, respectively.

SELECTION OF METHODS OF TESTS

A 3 by 6-in. cylinder has been maintained as the specimen used in the freezing-and-thawing tests because it is a convenient size, is about the smallest specimen in which $\frac{3}{4}$-in. max aggregate can be used, and because it occupies a comparatively small amount of space. It can be tested for compressive strength and elastic properties by the static method, and with inserts cast in its ends it can be easily measured for volume change. The specimens are frozen in water because freezing can be obtained more rapidly in this fashion, and it is evident that the results, as indicated by weight loss and expansion, are every bit as reliable as other methods.

Analysis of deterioration shows that more scaling and failure of the concrete through crumbling occurs when freezing is in water than when it is done in air. Weight loss is not a good criterion of failure for concrete frozen in air. Figure 2 compares concretes which have been frozen under the two conditions. Concrete of poor quality frozen in water usually fails by the entire specimen becoming soft and crumbly, and concrete of excellent quality may not begin to scale until after more than 200 cycles of the test. In these cases there is little

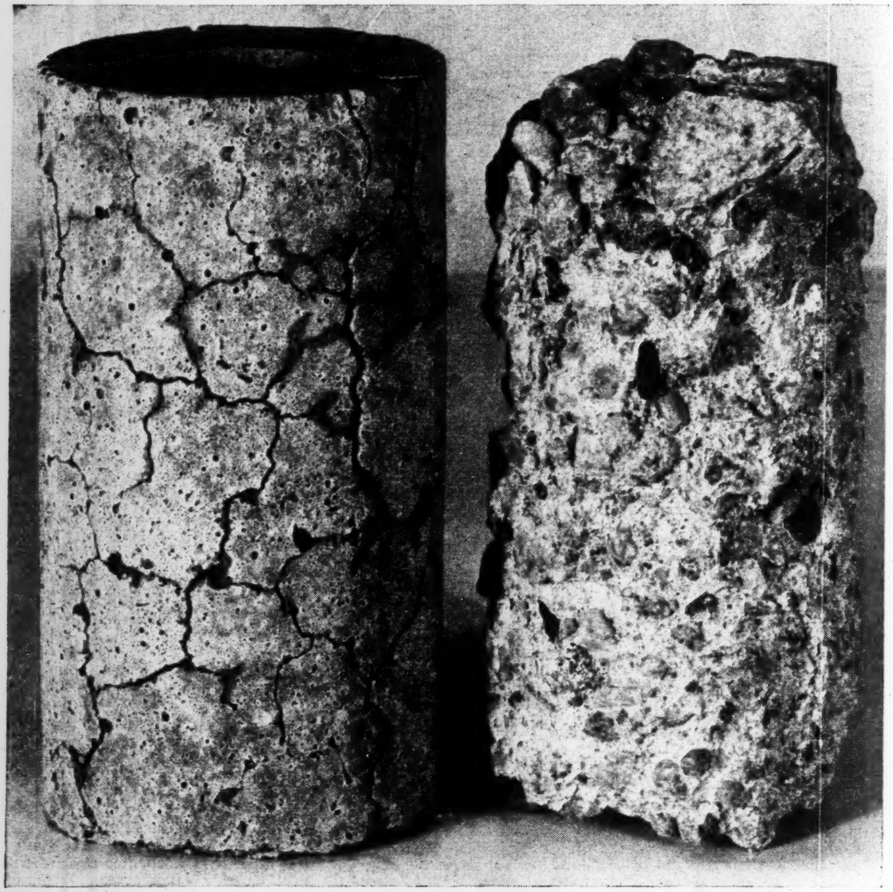
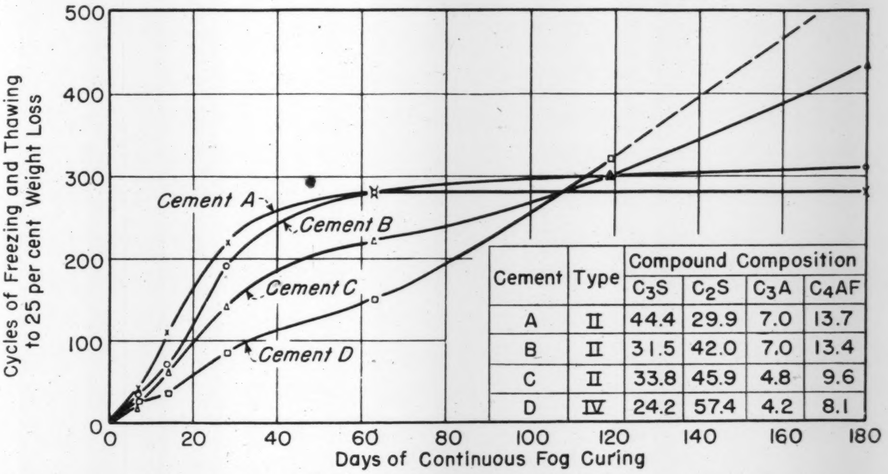


FIG. 2.—Typical Failures of Specimens Frozen in Air and Frozen in Water

correlation between weight loss and expansion during the early stages of the test. Generally the correlation is good at 25 per cent weight loss when failure of the concrete for practical purposes has occurred.



Cement	Type	Compound Composition			
		C_3S	C_2S	C_3A	C_4AF
A	II	44.4	29.9	7.0	13.7
B	II	31.5	42.0	7.0	13.4
C	II	33.8	45.9	4.8	9.6
D	IV	24.2	57.4	4.2	8.1

FIG. 3.—Type of Cement and Type of Curing Affects the Resistance of Concrete to Freezing and Thawing.

All aggregates are graded the same before incorporation in concrete so that the results can be compared with those obtained with other aggregates and so that variations, as far as possible, will be limited to the quality of the aggregate. A smaller percentage of sand is used in the air-entrained concretes than in the plain concretes as would be the case in practice. The aggregate is used in a dry state because it is easier to handle in this fashion and because it has been concluded that saturation only accelerates the failure of the concrete.

A comparatively low water-cement ratio of 0.51 by weight is used so that a comparatively high quality paste will be obtained and so that the failure will be less likely to occur in the paste at an early period. Although it is desirable to obtain the results as soon as practicable, it has been decided that the concrete should be cured for at least 28 days before starting the tests. Otherwise, the failure might occur in the paste and differences in the quality of the aggregate might not be indicated. Considerable variation can be obtained in the results of the freezing-and-thawing tests with the same aggregates and different cements. For that reason an attempt is made to obtain a uniform cement by blending nine Type II cements from mills in the Rocky Mountain area.

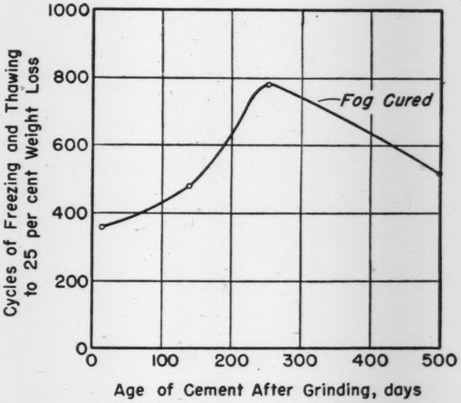


FIG. 4.—Age of Cement has an Effect on the Resistance of Concrete to Freezing and Thawing.

The freezing-and-thawing concrete specimens are compacted by internal vibration. This method of compaction was selected after it was demonstrated that there was not much difference in the resistance to freezing and thawing of the hand-rodded specimens and the vibrated specimens. Care must be taken, however, in vibrating the specimens, particularly when concretes contain entrained air, because it is quite easy to vibrate much of the air out of the concrete. Also, observations of failure of the specimens show that where the vibrator is held near the outside surface, signs of failure first appear near these points of greater vibration.

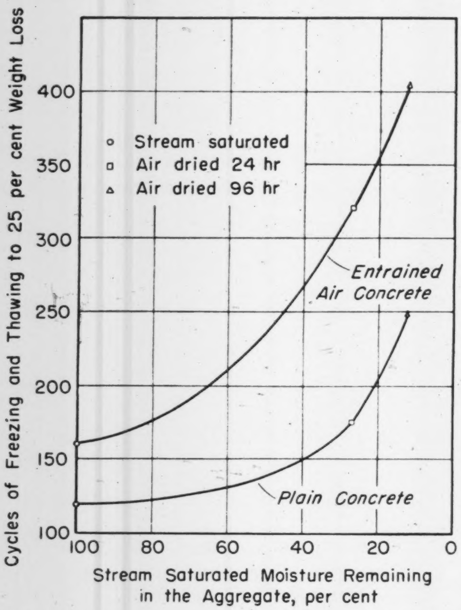


FIG. 5.—Drying of Aggregate Prior to Mixing Increases Durability of Concrete.

SOME FACTORS AFFECTING RESULTS

The resistance of concrete to freezing and thawing is greatly influenced by the type and brand of cement. Figure 3 shows the results obtained with different brands of cement and the same aggregate. For the purpose of evaluating an aggregate in an acceptance test, it is necessary to use the same cement in all mixes; otherwise, the test is quite likely to show the effect of the cement rather than the aggregate. However, when it is desired to know the actual performance of the aggregate used in the field structure it is desirable that it be tested with the cement which will be used on the job. The age of cement has an appreciable effect on the results obtained. Figure 4 shows how the resistance of concrete to freezing and thawing increased as the age of the cement after grinding increased up to an age of about 250 days. Although the trend was reversed and a lower resistance to freezing and thawing was obtained at 500 days nevertheless this age produced better results than did the freshly ground cement. This cement was stored in sealed metal drums in the comparatively dry atmosphere at Denver, Colo. This variation in cement with age and of different grinds received from the mill makes it very difficult to compare the results obtained at one time with those obtained at some other period.

The length of curing has a definite effect on the resistance of concrete, and concretes made with some cements show superior resistance after short curing periods as compared with those cured for longer periods. Also, concretes containing accelerators such as calcium chloride might show comparatively higher durability after short periods of curing because of higher early strengths developed.

The moisture content of the aggregate at the time of mixing has a great influence on the resistance of concrete to freezing and thawing, as indicated by the results shown in Fig. 5. These results were obtained with aggregates dug from the river and immediately placed in drums filled with water for shipment to the laboratory. Mixes were made with the aggregate as received in a saturated state, after being dried for 24 hr, and after being dried for 96 hr. The results obtained in these tests agree with the behavior of the concrete observed in the field in that the structures near the river where saturated aggregate is more likely to be used, shows poorer resistance to weathering.

The 3 by 6-in. specimens used in these tests are placed in tight-fitting rubber containers with only sufficient water in the container to cover the top of the specimen. Where the volume of the specimen is reduced by scaling, the cycles of freezing and thawing become progressively less severe as the volume of water in the tube increases. The degree of saturation of the concrete, the percentage of entrained air contained in the concrete, and other factors which have not been discussed may have important effects on the results.

CONCLUDING REMARKS

It is believed that it will be difficult to establish a standard criterion for freezing-and-thawing tests for the entire country because each apparatus has its own inherent characteristics and because small changes in the freezing temperature and test procedure produce variable effects on the results. Also, each cement will give slightly different results, and mixing, curing, and handling procedures will add to the variations obtained. However, it is believed that each laboratory can set its own standards for evaluating the results of this test, and we have complete confidence that when a concrete withstands 1000 cycles under our present procedure it will behave very satisfactorily under severe weathering conditions. We are also confident that a concrete which fails in less than 150 cycles should not be used under severe weathering conditions.

Acknowledgment:

The assistance of H. W. Brewer in compiling and reviewing data for this paper is gratefully acknowledged.

REFERENCES

(1) W. H. Price, prepared discussion of "Considerations Involved in the Making of Freezing and Thawing Tests of Concrete," *Proceedings*, Am. Soc. Testing Mats., Vol. 46, p. 1212 (1946).

(2) C. E. Wuerpel, "Laboratory Studies of Concrete Containing Air-entraining Admixtures," *Proceedings*, Am. Concrete Inst., Vol. 42, pp. 305–354 (1946).

(3) F. N. Wray, and H. J. Lichtefeld, "The Influence of Test Methods on Moisture Absorption and Resistance of Coarse Aggregate to Freezing and Thawing," *Proceedings*, Am. Soc. Testing Mats., Vol. 40, pp. 1007 (1940).

(4) B. G. Long, and H. J. Kurtz, "Effect of Curing Methods Upon the Durability of Concrete as Measured by Changes in the Dynamic Modulus of Elasticity," *Proceedings*, Am. Soc. Testing Mats., Vol. 43, p. 1051 (1943).

(5) C. A. Hughes, "The Durability of Cement Mortars: The Cement and Method of Testing Major Variables," *Proceedings*, Am. Soc. Testing Mats. Vol. 33, Part II, p. 511 (1933).

(6) H. W. Brewer, and R. W. Burrows, "Coarse-ground Cement Makes More Durable Concrete," *Journal*, Am. Concrete Inst., No. 5, Vol. 22, pp. 353–360, Jan., 1951.

(7) W. E. Gibson, "Significance of Soundness Tests of Aggregates," *Proceedings*, Highway Research Board, Vol. 21, pp. 283–287 (1941).

(8) "Progress Report of Committee on Durability of Concrete," *Proceedings*, Highway Research Board, Vol. 24, pp. 174–201 (1944).

(9) R. E. Bollen, "Durability of Portland Cement Concrete Determined by Primary Directional Freezing and Uniform Thawing," *Proceedings*, Highway Research Board, Vol. 28, pp. 218–229 (1948).

(10) C. E. Wuerpel, E. O. Rexford, "The Soundness of Chert as Measured by Bulk

Specific Gravity and Absorption," *Proceedings*, Am. Soc. Testing Mats., Vol. 40, p. 1021 (1940).

(11) M. O. Withey, "Factors Affecting the Resistance to Freezing and Thawing of Vibrated Concrete Made of Crushed Dolomite," *Proceedings*, Am. Concrete Inst., Vol. 35, pp. 553–560 (1939).

(12) E. R. Dawley, "Correlation between Rapid Immersion and Standard Four Cycle per Day Freezing and Thawing Tests," *Proceedings*, Am. Concrete Inst., Vol. 37, pp. 157–160 (1941).

(13) W. C. Hansen, "Influence of Sands, Cements, and Manipulation upon the Resistance of Concrete for Freezing and Thawing," *Proceedings*, Am. Concrete Inst., Vol. 39, pp. 105–123 (1943).

(14) Stanton, Walker, "Freezing and Thawing Tests Made with Different Aggregates," *Proceedings*, Am. Concrete Inst., Vol. 40, pp. 573–577 (1944).

DISCUSSION

MR. A. D. CONROW.[1]—In the case of the automatic freezing-and-thawing apparatus, is there much chance of thermal shock when the warming water is turned in to thaw the specimens in the automatic apparatus?

MR. W. H. PRICE (*author*).—The thermal shock in the automatic apparatus is the same as that in the manually operated apparatus, which was previously used in our laboratories. In each case, specimens were frozen for approximately 1½ hr and thawed for 1½ hr. In the case of the manually operated apparatus, the specimens were moved by hand from the thawing tank to the warm fluid tank and fewer cycles a day were obtained with this manually operated apparatus. Because a larger number of cycles are obtained with the automatic apparatus in less time, it is natural to assume that this apparatus will cause the specimen to fail sooner. This is not the case, however, and it is indicated that the failure in fewer cycles in the manually operated apparatus was due in part to absorption which occurred while the specimens were emersed in the warm tank at night and on weekends. This difference due to absorption was not great, and, as has been explained in the paper, a slight lowering of the freezing temperature has a much greater effect on the results.

MR. P. H. KAAR.[2]—I should like to ask whether the authors have ever established a correlation between soundness tests of the individual aggregate to soundness tests of the same aggregates mixed in concrete?

MR. PRICE.—The concrete made with Kimball aggregate failed in only 30 cycles. This aggregate passed all the standard tests for specific gravity, sodium sulfate, and Los Angeles abrasion very satisfactorily. An examination of the aggregate indicated that it was of good physical quality. This aggregate contains constituents which will react with alkalies in the cement, but even if a low-alkali cement is used, the concrete will fail in a comparatively few cycles of freezing and thawing. This particular aggregate has also had a very poor service record. There are many other aggregates which have passed the requirements of the standard physical tests and also produced durable concrete such as the Grand Coulee aggregate which was described in our paper.

MR. C. U. PIERSON, JR.[3]—How would one correlate the performance of freezing-and-thawing tests *versus* the performance of concrete in areas where there is no freezing and thawing but there is still a lack of durability?

MR. PRICE.—A wetting-and-drying

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[1] Research Engineer, Ash Grove Lime and Portland Cement Co., Chanute, Kans.

[2] Civil Engineer, U. S. Bureau of Mines, Schuylkill Haven, Pa.

[3] Chief Research Assistant, Marquette Cement Manufacturing Co., Des Moines, Iowa.

test has been used for evaluating materials from southern portions of our country. The freezing-and-thawing tests are used to indicate the over-all soundness or durability of the concrete, but these results have not been correlated with field performance in the southern states. We did make an attempt in this paper at such a correlation for concretes places in the northern states.

MR. R. R. LITEHISER.[4]—I should like to have the authors elaborate just a little bit on whether there is any correlation between the results of their tests on an aggregate—a coarse aggregate, let us say and the results of freezing-and-thawing-test and the sodium sulfate soundness test.

MR. PRICE.—We are now preparing a paper which will attempt to correlate the freezing-and-thawing tests and the sodium sulfate soundness test. In recent months all aggregates submitted for acceptance have been subjected to both tests. It is indicated that there might be some correlation between the results of the two tests.

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[4] Chief Engineer, Testing and Research Lab., Department of Highways, Columbus, Ohio.

PERFORMANCE OF AUTOMATIC FREEZING-AND-THAWING APPARATUS FOR TESTING CONCRETE*

By Stanton Walker[1] and Delmar L. Bloem

Synopsis

The design features, operation, and performance characteristics of an automatic machine for subjecting concrete specimens to freezing and thawing are described. A brief discussion is presented of the factors which influence results of freezing-and-thawing tests and which must be taken into consideration in designing such equipment.

The ability of the apparatus to evaluate the influence of various factors on resistance of concrete to freezing and thawing is demonstrated by data from several investigations. Limited data are presented comparing the severity of freezing in air with freezing in water and comparing the performance of two automatic machines of different types.

The described machine has been found to perform satisfactorily during two years of operation. It combines the desirable features of automatic control and reasonable speed of testing with a high degree of flexibility of operation. Since it can be used to freeze specimens in either air or water at different rates and to different temperatures, it is particularly well adapted to studying the factors which influence freezing-and-thawing test results.

Freezing-and-thawing tests have been shown to be useful in studies of the durability of concrete. Methods and equipment are almost as numerous as the organizations making the tests. Much valuable information has been developed, but there has been no standardization of procedures—and that, in the light of present information, is as it should be.

The failure to arrive at a method generally enough acceptable to justify standardization comes in part from the fact that different procedures give different results and that their relationship to what happens in nature has not been evaluated. Another factor, and perhaps a more important one, is the failure to secure satisfactory reproducibility of results among different laboratories and, sometimes, in the same laboratory. Minor variations in the several phases of the test, from molding and curing through the actual freezing and thawing, frequently affect the results enough to obscure their significance.

The objective of testing concrete by freezing and thawing is to produce an exposure sufficiently severe to distinguish among concretes of different qualities. That is to say, the test must cause deterioration in concrete which, because of inferior materials, poor proportions, unsatisfactory curing conditions or other reasons, has less than adequate resistance to freezing and thawing. On the other hand, the exposure must not be excessively severe; it must not destroy all concrete so rapidly that no distinction is made as to degree of durability. A compromise must be found in a procedure as severe as consistent with securing a significant spread in results between resistant and non-resistant concrete in a reasonable period of time.

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*Presented at the Fifty-fourth Annual Meeting of the Society, June 18–22, 1951.

[1] Director of Engineering and Assistant Director of Engineering, respectively, of the National Sand and Gravel Assn. and the National Ready Mixed Concrete Assn., Washington, D. C.

Much progress has been made in the development of equipment and procedures which greatly improve reproducibility of tests within a given laboratory and expedite the securing of results. The different approaches involve slow or fast cycles, freezing in air or in water, freezing and thawing to different temperatures, etc. No one type of cycle has been proven to be superior to another. A number of automatic machines have been developed (**40, 42, 43, 48**).[2]

The development of freezing-and-thawing tests as a controlled procedure began in the early 1920's. Withey, Scholer and Lang (**1, 10, 11**) were among the pioneers, and only slightly later much work was done by the Portland Cement Assn., the Bureau of Public Roads, the Missouri Highway Department, and others. There is appended a selected list of the literature which it is believed will call the reader's attention to the principal sources of information pertinent to the development of the test method itself.

No attempt is made in this paper to summarize or correlate results of past work. Out of that work has grown the realization that numerous factors, many of them difficult of control, affect the results. Among the principal of these are the degree of saturation of the concrete, the rate of freezing, the rate of thawing, the temperature to which frozen, the temperature to which thawed, whether frozen in air or in water, as well as many others.

Both the National Sand and Gravel Assn. and the National Ready Mixed Concrete Assn have an outstanding interest in the development of procedures for studying the durability of concrete. Since 1940, researches into freezing and thawing have represented a major portion of the activities of the Research Laboratory jointly sponsored by the two Associations at the University of Maryland (**30, 36, 37, 51, 52, 53, 54, 55, 56, 57**). Automatic equipment, differing significantly from others currently in use and capable of producing 7 to 10 cycles of freezing and thawing per day has recently been developed. It is the purpose of this paper to describe the essential features of that equipment and to furnish information concerning its performance. A limited amount of data are included comparing its performance with that of another automatic machine.

DESCRIPTION OF APPARATUS

A schematic drawing of the freezing and thawing apparatus is shown in Fig. 1. It consists essentially of an insulated, watertight chamber equipped with refrigeration coils, over a tank which contains the water for thawing. The specimen chamber is cooled by means of a 5 hp compressor using freon No. 12 as the refrigerant. The thawing water tank is equipped with six 1000-w electric immersion heaters to restore the heat used in thawing the concrete specimens.

During the freezing cycle, air in the specimen chamber is circulated by two 12-in. fans powered by $\frac{1}{8}$-hp motors, to insure rapid heat transfer and uniform temperature conditions throughout the chamber. The concrete specimens are supported on a false bottom of steel grating to permit free circulation of air or water to all surfaces. A 50 gal. per min centrifugal pump circulates the thawing water through the specimen chamber during the thawing phase of the cycle. The equipment was constructed and installed by the Carrier Division of the United Clay Products Corp. in accordance with specifications and schematic plans preparéd by the authors.

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[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p.1132.

The apparatus was originally designed to freeze the specimens in air and thaw them in water. However, provision has also been made to permit both freezing and thawing in water. This is accomplished by placing the specimens in watertight metal containers and requires no modification in operation except minor adjustment in freezing temperature and length of cycle, as discussed later.

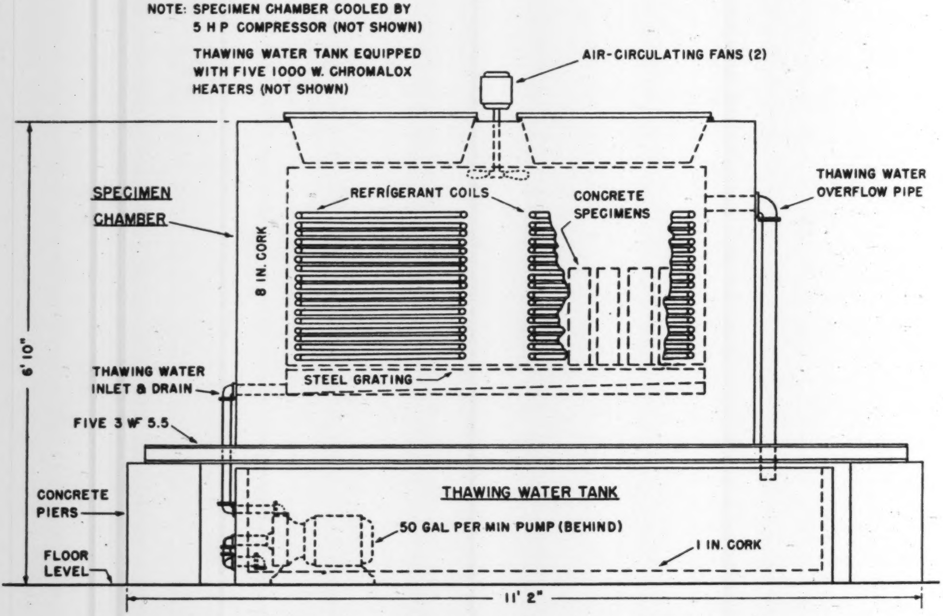
NOTE: SPECIMEN CHAMBER COOLED BY
5 H P COMPRESSOR (NOT SHOWN)
THAWING WATER TANK EQUIPPED
WITH FIVE 1000 W. CHROMALOX
HEATERS (NOT SHOWN)
AIR-CIRCULATING FANS (2)
SPECIMEN
CHAMBER
REFRIGERANT COILS
CONCRETE
SPECIMENS
THAWING WATER
OVERFLOW PIPE
8 IN. CORK
6' 10"
THAWING WATER
INLET & DRAIN
STEEL GRATING
FIVE 3 WF 5.5
CONCRETE
PIERS
THAWING WATER TANK
50 GAL PER MIN PUMP (BEHIND)
1 IN. CORK
FLOOR
LEVEL
11' 2"

FIG. 1.—Schematic Drawing of Automatic Freezing-and-Thawing Apparatus.

When freezing is in air, the compressor is regulated thermostatically to reduce the air temperature in the specimen chamber to 0 F and maintains that temperature for a sufficient period to insure that the specimens (normally 3 by 4 by 16 in.) have reached 0 F throughout. The fans operate continuously during this time. At the end of the freezing phase of the cycle, the compressor and fans are cut off by a time-delay relay switch and, at the same time, the centrifugal pump is started.

Water at approximately 40 F is pumped from the thawing water tank, fills the specimen chamber and returns through the overflow pipe, circulating continuously for a sufficient time to thaw the specimens and increase their internal temperature to 40 F. The quantity of thawing water and heating capacity of the immersion heaters are such that the water temperature is reduced less than 2 F during the thawing cycle and is restored to its original temperature before the next thawing cycle. At the close of the prescribed thawing period, a second time-delay relay switch turns off the pump and actuates a third timer which permits a sufficient delay to allow the thawing water to drain back through the pump into the thawing water tank. At the end of the drain period, the compressor is again started, automatically, for the beginning of the next cycle.

When specimens are frozen in water, the cycle is essentially the same as described above except that the air temperature during freezing is reduced to −10 F and the length of the thawing period is increased. These changes are necessary because of the additional water to be frozen and thawed.

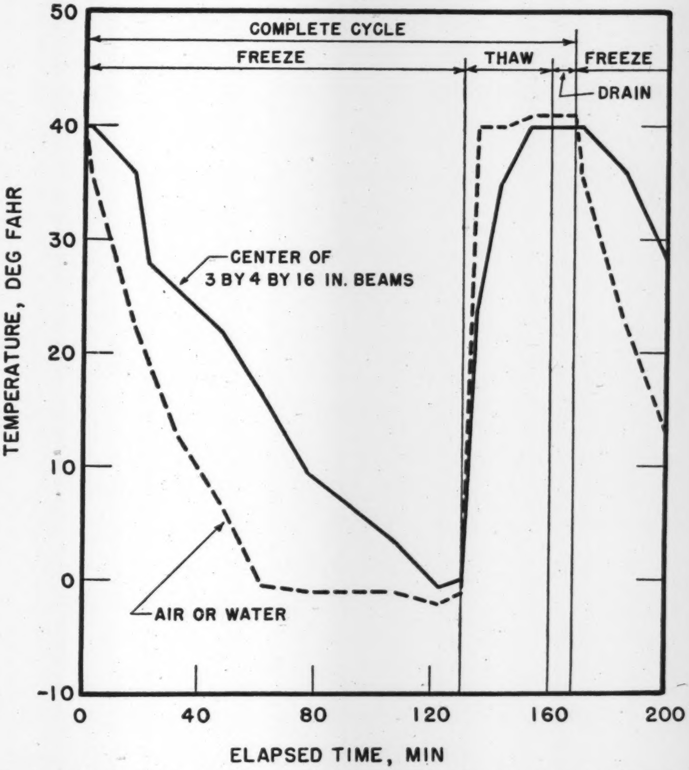

Fig. 2.—Typical Cycle in Automatic Freezing-and-Thawing Apparatus. (Specimens Frozen in Air, Thawed in Water.)

Performance Characteristics

The typical time-temperature cycle when specimens are frozen in air is shown in Fig. 2. It can be seen that the air temperature is reduced to 0 F in about 1 hr of the freezing cycle and that, within 2 hr, the centers of the beams have also reached 0 F. An additional 10 min is allowed during which the specimens remain at 0 F, making the total length of the freezing cycle 2 hr and 10 min.

The ambient temperature quickly rises to 40 F at the beginning of the thawing cycle and remains constant within about 1 deg during the remainder of the thawing period. The centers of the specimens reach 40 F within less than 25 min and remain at that temperature during the remainder of the 30-min thawing cycle.

Temperature in the specimen chamber remains essentially constant during the 8-min period allowed for the water to drain back into the thawing water tank. Specimen surfaces are still wet at the end of this interval, and, since the air temperature falls below freezing in less than 10 min of the next freezing cycle, there is little opportunity for moisture to be evaporated from the concrete during the air freezing.

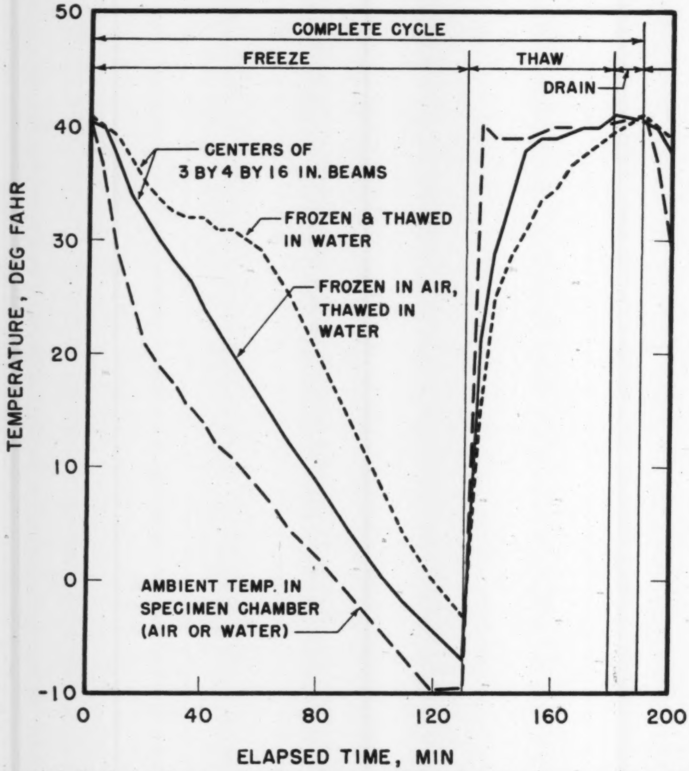

Fig. 3.—Typical Cycle in Automatic Freezing-and-Thawing Apparatus. (Two Types Exposure Simultaneously.)

When freezing is in air, each complete cycle requires 2 hr and 48 min. Once each day the apparatus is turned off to permit weight and dynamic modulus of elasticity determinations on the specimens. This is done at the end of a thawing cycle, and the specimens in the chamber are kept immersed by closing the valve to the pump. With this delay taken into account, the apparatus still produces slightly more than 8 complete cycles per day when freezing is in air. Since the freezing chamber will hold 50 specimens, the total capacity is approximately 400 beam-cycles per day. A larger number of cycles could be secured by reducing the freezing chamber to a temperature lower than 0 F or by permitting a temperature higher than 0 F in the specimens.

For freezing in water, the specimens are immersed in water-tight metal containers $3\frac{1}{8}$ by $4\frac{1}{8}$ by $16\frac{1}{4}$ in. Time-temperature relationships for this cycle are illustrated in Fig. 3, both for specimens frozen in water and for specimens frozen simultaneously in air with the chamber containing half of each.

In order to reduce the additional time required to freeze and thaw the greater quantity of water, the air temperature is regulated to fall to −10 F rather than to 0 F. This results in a reduction in the length of time for the specimens in air to reach 0 F from the 2 hr required in the normal cycle to about $1\frac{3}{4}$ hr. The specimens in water reach 0 F in about two hr, and the total length of freezing cycle is maintained at the 2 hr, 10 min used in the air-freeze cycle.

TABLE I.—TEMPERATURE VARIATIONS IN SPECIMEN CHAMBER (FREEZING IN AIR; THAWING IN WATER).

Elapsed Time from Beginning of Freeze Cycle, min	Temperature, deg Fahr[a]					
	Coil 1	Coil 2	Coil 3	Coil 4	Coil 5	Coil 6
Freezing Cycle (Air Temperatures)						
0	41	40	41	42	42	42
15	21	20	20	30	17	42
30	13	12	11	16	9	42
45	5	4	3	8	1	42
60	0	0	−1	4	−2	42
75	−1	−2	−2	2	−3	42
105	−2	−3	−2	0	−3	42
130	−2	−2	−3	−1	−2	42
Thawing Cycle (Water Temperatures)						
140	40	39	40	41	39	41
150	41	40	41	42	42	41
160	41	40	41	42	42	41
End of Drain Cycle (Air Temperature)						
168	41	40	41	42	42	42

[a] Temperatures measured by means of resistance coils located at various positions in chamber, as follows:
Coil 1—Top center of right compartment.
Coil 2—Mid-height center of right compartment.
Coil 3—Bottom center of right compartment.
Coil 4—Mid-height extreme left side of right compartment.
Coil 5—Mid-height extreme right side of right compartment.
Coil 6—In thawing-water tank.

The thawing time for specimens frozen in water is increased from about 30 min to 50 min with the result that the total length of a complete cycle is increased to about 3 hr, 10 min. The capacity of the machine is, therefore, reduced only from about 8 to 7 complete cycles per day including the necessary shutdown time for taking readings.

The reproducibility of the temperature curves in both types of cycle has been found to be good for specimens in various locations in the specimen chamber and for tests made at different times. Table I gives temperatures measured in the thawing water tank and simultaneously at different points in the specimen chamber during a typical cycle.

The only major difficulty so far encountered has been the high mortality rate of the metal containers used for freezing specimens in water. Slight increases in the thickness of water film between specimen and container, resulting from the surface erosion of the concrete, have been found to cause progressive expansion and eventual rupture of the container because of expansion of the water upon freezing. Recent experience indicates that a rubber diaphragm cemented over a slit in the side of the container will eliminate this difficulty.

Test Results

The ultimate measure of worth of various types of freezing-and-thawing equipment is their ability to distinguish among concretes of different quality. Figures 4 through 7 show test results which provide such information for this machine. In all cases, the concrete for which data are given was of average richness ($5\frac{1}{2}$ sacks of cement per cu yd) and average consistency (3 to 4-in. slump). For the most part, tests were made of both normal and air-entraining concrete (4 to 5 per cent air), as indicated on the diagrams. Each curve represents the average for specimens from at least 3 batches of concrete mixed on different days.

Figure 4 shows data for concretes made with three coarse aggregates. By conventional standards, aggregate No. 1, a quartz gravel, was of excellent quality, being characterized by very low absorption and sulfate soundness losses and the virtual absence of particles classed as deleterious. Aggregate No. 3 was of intermediate quality, having moderate absorption (1.4 per cent), a 5-cycle sodium sulfate soundness loss of 7.6 per cent and a total of 13 per cent of particles classed as deleterious. Aggregate No. 2 was of poorer quality, having relatively high absorption (2.5 per cent), a 5-cycle sodium sulfate soundness loss of 12.8 per cent and 19 per cent of particles classed as deleterious.

As shown in Fig. 4, the freezing-and-thawing test gave results consistent with other measures of the quality of the three aggregates. Both normal and air-entraining concrete containing the good quality aggregate (No. 1) withstood 250 cycles without appreciable reduction in dynamic modulus, when the test was discontinued. Aggregate No. 3 gave better performance than No. 2 in both normal and air-entraining concrete, but both aggregates showed considerably less resistance than No. 1. Although the performances of aggregates Nos. 2 and 3 were markedly improved by air entrainment, the test was sufficiently severe to cause deterioration and distinguish among the different aggregates in the air-entraining concrete.

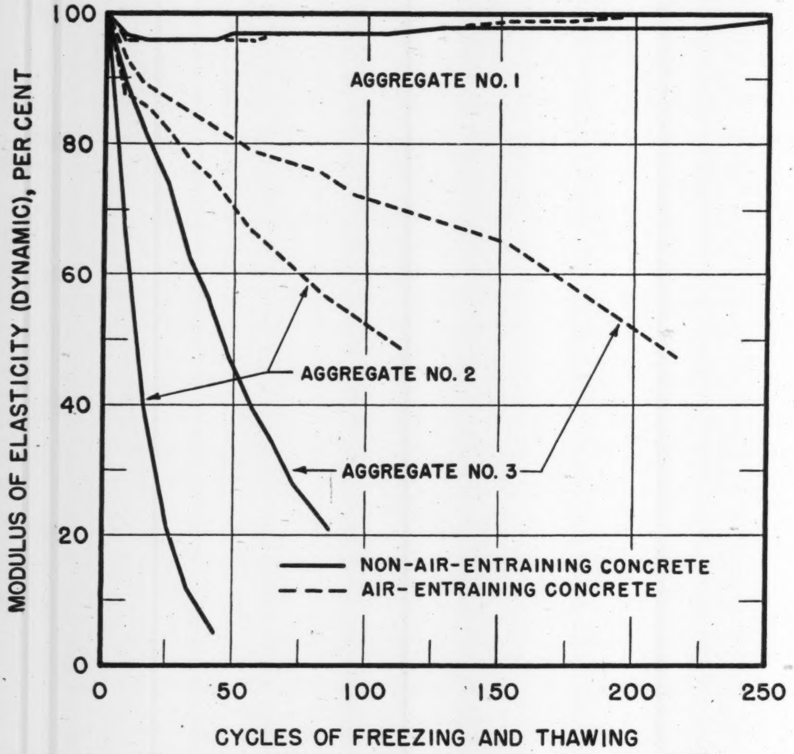

FIG. 4.—Results of Freezing-and-Thawing Tests of Concrete Containing Different Aggregates (Series J44).

Figure 5 shows results of tests made to determine the effect of certain processing applied to a coarse aggregate on its resistance to freezing and thawing in concrete. The test successfully distinguished between the treated and untreated aggregates. The performance of gravel No. 2 in both normal and air-entraining concrete was appreciably improved by the processing. For all gravels, performance was improved by the use of air entrainment but not to the extent that it could be said to have insured good performance of the inferior gravel.

Figures 6 and 7 illustrate comparative tests made to determine the relative severity of freezing in air and in water. The data indicate that, as done in this apparatus, freezing and thawing in water is more severe than freezing in air and thawing in water.

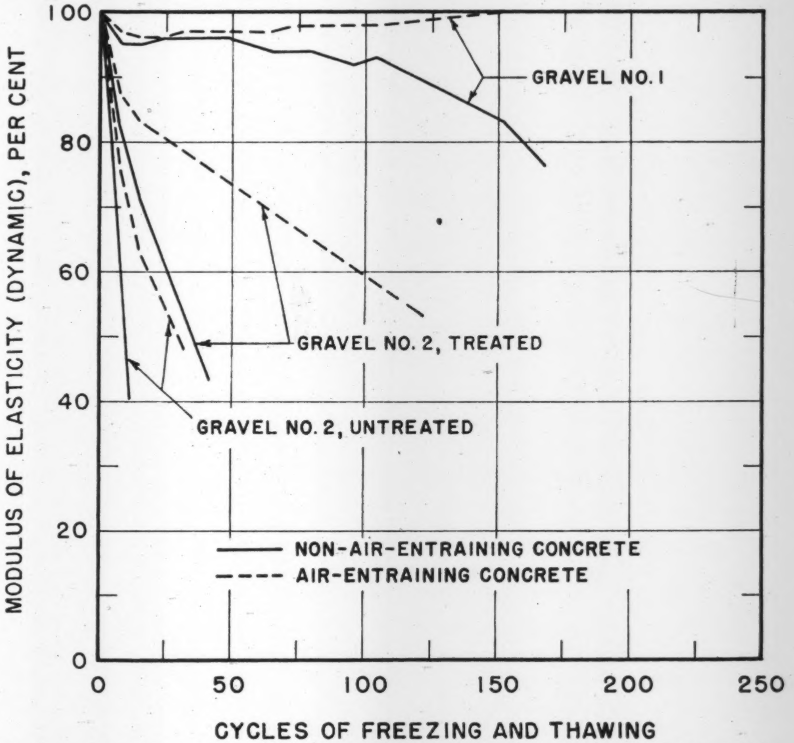

Fig. 5.—Results of Freezing-and-Thawing Tests of Concrete Containing Different Coarse Aggregates (Series 124).

Figure 6 shows test results for air-entraining concrete containing an untreated gravel of high soft stone content and the same gravel treated mechanically to remove soft particles, compared with stock quartz gravel. For all three concretes, failure occurred more quickly when specimens were frozen and thawed in water than when they were frozen in air and thawed in water. The three aggregates line up in the same order for either test method. However, effect of the processing appears to be more clearly marked in the air-freeze cycle.

Figure 7 shows results of tests on both normal and air-entraining concrete containing four different fine aggregates but the same high-quality stock quartz gravel. In this case, neither type of cycle produced appreciable reduction in dynamic modulus of any of the air-entraining concrete in 250 cycles of freezing and thawing. The curves for all conditions of air entraining concrete are contained in the cross-hatched area as are the curves for normal concrete for specimens made with sands 2, 3, and 4

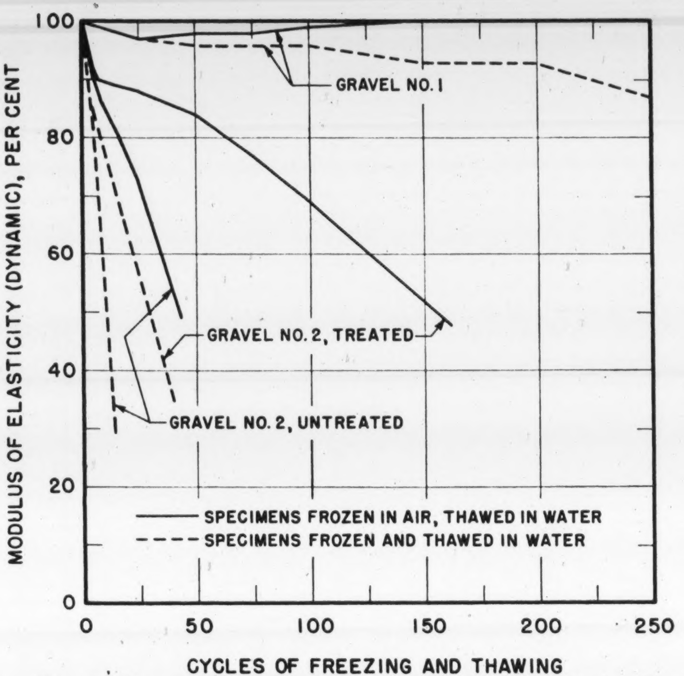

FIG. 6.—Comparison of Two Types of Freezing-and-Thawing Exposures in Same Apparatus (Series 136).

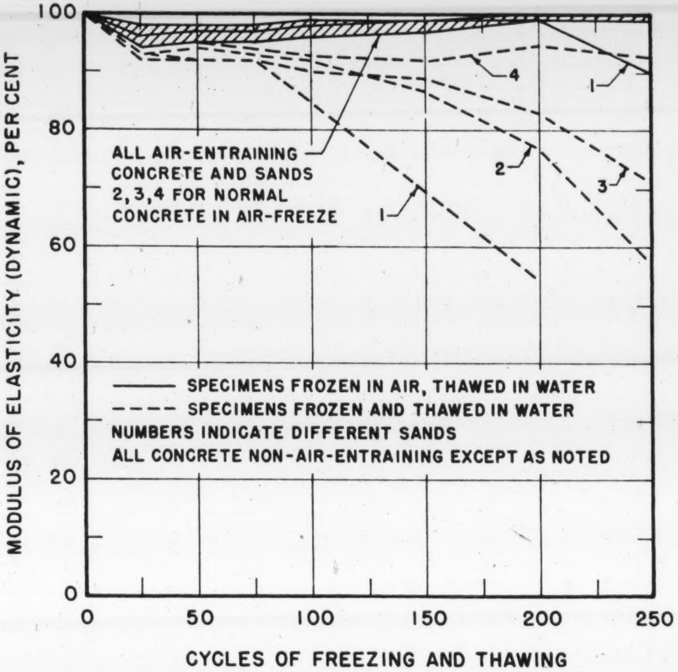

FIG. 7.—Comparison of Two Types of Freezing-and-Thawing Exposures in Same Apparatus (Series 132).

frozen in air. In the non-air-entraining concrete, the air-freeze, water-thaw cycle caused a slight reduction in dynamic modulus of concrete containing sand No. 1, but virtually none for sands Nos. 2, 3, and 4. The water-freeze cycle, on the other hand, produced a wide spread in results for the non-air-entraining concretes and indicated that the sands were increasingly resistant in the order 1, 2, 3, 4. These results do not correlate well with losses in 10-cycle-sodium-sulfate tests on these sands which, in the order indicated, were: 1.4; 6.7; 7.9; and 9.7 per cent—the reverse of what might have been expected. However, none of the sands gave results generally considered unacceptable, and there were other conditions, principally surface texture, and perhaps also grading, which may account for the trend. The two freezing-and-thawing test methods correlated to the extent that sand No. 1 gave the poorest performance in both cases.

In these comparative tests there has been noted one consistent difference in the type of breakdown produced by the two testing procedures. Specimens frozen and thawed in water consistently develop surface scaling, whether or not there is any appreciable reduction in dynamic modulus. This type of disintegration is almost entirely absent in the air-freeze, water-thaw cycle even for specimens having large reductions in dynamic modulus.

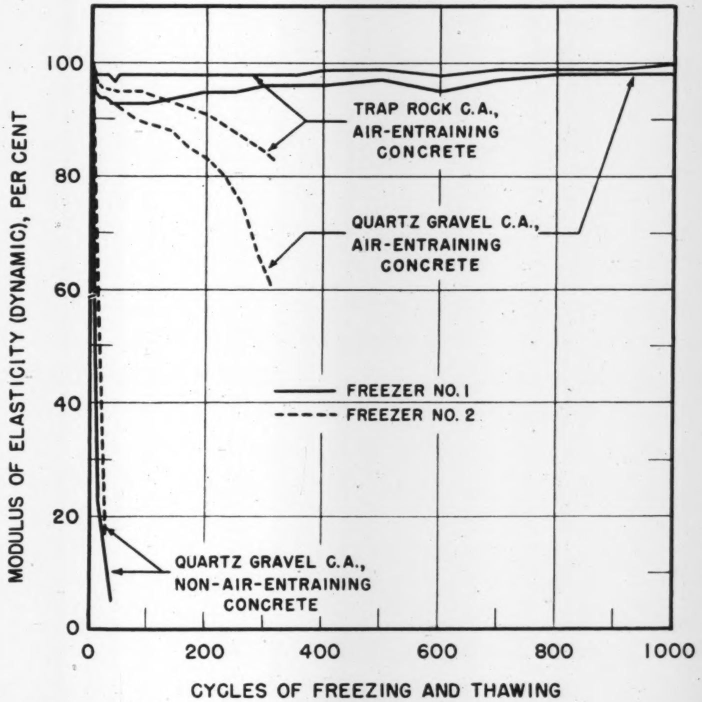

FIG. 8.—Comparison of Two Types of Freezing-and-Thawing Cycles (Series 121).

COMPARISON WITH OTHER EQUIPMENT

Through cooperation of the Corps of Engineers, U. S. Army, comparative freezing-and-thawing tests were made in the equipment described in this report, with specimens frozen in air and thawed in water, and in the automatic apparatus at the Concrete Research Division, Waterways Experiment Station, Jackson, Miss., in which specimens were frozen and thawed in water. The latter equipment, described elsewhere in the literature (40, 43), produces 12 cycles per day of freezing to 0 F and thawing to about 40 F of $3\frac{1}{2}$ by $4\frac{1}{2}$ by 16-in. beams immersed in water in metal jackets.

In this cooperative study every effort was made to hold all variables constant except the type of freezing-and-thawing cycle. All concrete was mixed and specimens molded in the laboratory at the University of Maryland. Three types of concrete were included: non-air-entraining concrete containing a good quality quartz gravel and air-entraining concretes containing the quartz gravel and a good quality crushed trap rock. The same fine aggregate, a good quality siliceous sand, and the same cement, a blend of equal amounts of four brands, were used throughout. All concrete contained $5\frac{1}{2}$ bags of cement per cu yd and had a slump of about 3 to 4 in.

Three batches of each of the three types of concrete were mixed on different days. Six $3\frac{1}{2}$ by $4\frac{1}{2}$ by 16-in. beams were molded from each batch, 3 for test at the University of Maryland and 3 for test at the Waterways Experiment Station. All specimens were kept in the molds under damp burlap for two days, after which they were stripped and cured in a standard moist room at 70 F for 10 days. At the age of 12 days all specimens were packed tightly in saturated sawdust in galvanized iron containers and the containers sealed. The 27 beams for test at the Waterways Experiment Station were shipped in these containers while the 27 companion specimens were retained in their containers at room temperature in

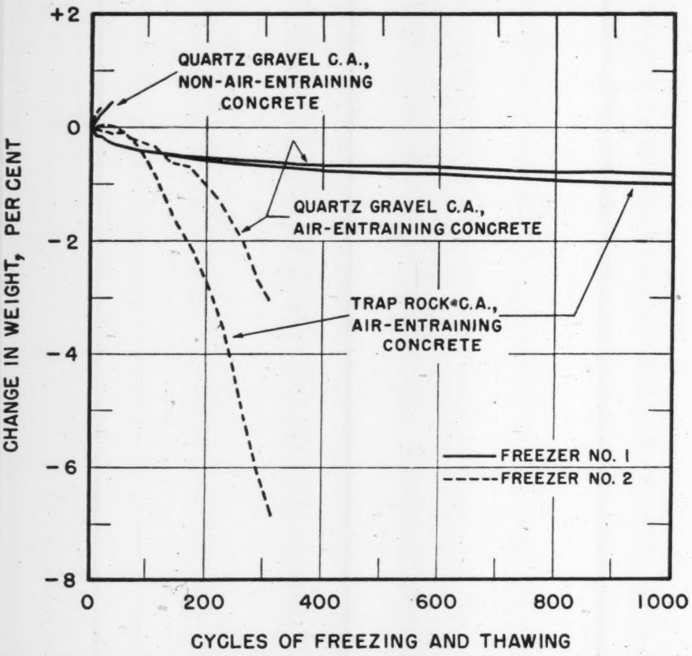

FIG. 9.—Changes in Weight of Concrete Specimens During Freezing and Thawing (Series 121).

the University of Maryland laboratory. At the age of 31 days, specimens in both laboratories were unpacked and immersed in saturated lime water for 7 days before being subjected to freezing and thawing. Measurements of weight changes during this 38-day period prior to freezing and thawing indicated good uniformity of moisture condition between companion specimens in the two laboratories and a rather high degree of saturation of all concrete.

Changes in weight and dynamic modulus of elasticity were determined at frequent intervals in both laboratories as a measure of deterioration during freezing and thawing. The test results are shown in Figs. 8 and 9. Freezer No. 1 indicates the equipment at the University of Maryland and freezer No. 2 the apparatus at the Waterways Experiment Station. It can be seen in Fig. 8 that the non-air-entraining concrete failed so rapidly in both exposures that no significant difference between test methods is discernible. This rapid failure probably may be attributed, at least in part, to the highly saturated condition of the concrete.

In the air-entraining concrete, on the other hand, marked differences between the two types of cycle were developed. In the equipment which froze in air and thawed in water (freezer No. 1) there was virtually no reduction in dynamic modulus of the air-entraining concrete in 1000 cycles of freezing and thawing. In the more rapid cycle of freezing and thawing of immersed specimens (freezer No. 2), 300 cycles reduced the dynamic modulus of air-entraining concrete containing the trap rock to about 83 per cent and the quartz gravel to about 60 per cent.

For the air-entraining concrete, the same difference in type of disintegration, previously noted for air and water freezing, was produced by the two types of apparatus. This is illustrated in Fig. 9 where changes in weight during freezing and thawing are shown. In the cycle in which specimens were frozen in air, there was only a very slight reduction in weight of the air-entraining concrete. This presumably was due principally to loss of moisture since there was no evidence of surface erosion from any of the specimens. In the water-freeze cycle there was severe scaling of mortar from the beam surfaces resulting in a weight loss of about 3 per cent for the quartz gravel concrete and 7 per cent for the trap rock concrete.

It is interesting to note that, on the basis of weight loss, the comparative performance of the two types of aggregate in the water-freeze cycle is opposite to that based on loss in dynamic modulus. The trap rock concrete, which had the lower reduction in dynamic modulus, showed a considerably greater loss in weight than the quartz gravel concrete which had the greater reduction in dynamic modulus.

Conclusion

Experiences with the automatic freezing-and-thawing apparatus described in this paper, as well as with manually operated equipment used since 1940, suggest the following comments:

1. The automatic apparatus is simple, dependable in operation and readily maintained.

2. The apparatus, although designed for freezing in air, can be adapted to freeze specimens either in air or water, or both at the same time.

3. A wide range in results has been produced for concretes of different qualities. General knowledge of the service records of similar concretes affords favorable evidence that test results are indicative of performance in service. However, the problem of correlating

actual resistance to weathering with resistance to this test, or any other freezing-and-thawing test, remains a formidable one.

4. A significant difference between results of freezing in water and in air is that the first causes marked surface scaling and erosion while the second results in substantially no visible effects until the specimen has progressed far toward failure. In the cooperative tests with the Corps of Engineers, the trap rock concrete showed the greater loss in weight and the lesser loss in dynamic modulus as compared with the quartz gravel concrete.

5. Freezing in water is more severe than freezing in air. These tests do not afford evidence to permit the suggestion that one procedure is more significant than the other.

6. The performance of 7 to 10 cycles per day, depending upon procedure, permits securing of test results within a reasonable period of time, from 200 to 300 cycles generally being adequate to show significant differences among the several concretes.

7. The apparatus described seems to be particularly adaptable to studying various freezing-and-thawing cycles.

REFERENCES

1928

(1) C. H. Scholer, "Some Accelerated Freezing and Thawing Tests," *Proceedings*, Am. Soc. Testing Mats., Vol. 28, Part II, p. 472 (1928).

1929

(2) F. H. Jackson, "Needed Research on Mineral Aggregates," *Proceedings*, Am. Soc. Testing Mats., Vol. 29, Part II, pp. 853–861 (1929).

1930

(3) H. F. Gonnerman and G. W. Ward, "Determining the Suitability of Aggregates for Concrete Pavements," *Proceedings*, 6th Annual Convention, Association of Highway Officials of the North Atlantic States, pp. 136–159, (1930).

(4) F. C. Lang and C. A. Hughes, "Discussion on Relation Between Durability of Concrete and Durability of Aggregates," *Proceedings*, Highway Research Board, Vol. 10, p. 113 (1930).

(5) C. H. Scholer, "Durability of Concrete," *Proceedings*, Highway Research Board, Vol. 10, p. 142 (1930).

1931

(6) W. J. Emmons, "The Effect of Soft Particles of Coarse Aggregate on the Strength and Durability of Concrete," *Circular No. 6*, Nat. Sand and Gravel Assn. (1931).

(7) A. T. Goldbeck, "Freezing Tests on Mortar and Concrete," *Crushed Stone Journal*, Vol. 7, No. 4, April, 1931, p. 5.

(8) F. H. Jackson, "Weathering Test Procedures for Concrete," *Proceedings*, Am. Soc. Testing Mats., Vol. 31, Part II, pp. 789–794, (1931).

(9) F. H. Jackson, "Resistance of Concrete to Frost Action as Affected by Water-Cement-Ratio," *Proceedings*, Highway Research Board, Vol. 11, Part 1, pp. 227–237 (1931).

(10) F. C. Lang and C. A. Hughes, "Accelerated Freezing and Thawing as a Quality Test for Concrete Aggregates," *Proceedings*, Am. Soc. Testing Mats., Vol. 31, Part II, p. 435 (1931).

(11) M. O. Withey, "Some Long Time Tests of Concrete," *Journal*, Am. Concrete Inst., February, 1931; *Proceedings*, Vol. 27, p. 547.

1932

(12) A .T. Goldbeck, "Freezing and Thawing Tests of Concrete Made with Stone-Sand Fine Aggregate," *Crushed Stone Journal*, Vol. 8, No. 7, Nov.–Dec., 1932, pp. 11–16.

(13) F. H. Jackson and George Werner, "The Resistance of Concrete to Frost Action," *Public Roads*, Vol. 13, No. 2, April, 1932, pp. 32–38.

(14) C. H. Scholer and A. E. Stoddard, "Proposed Method of Testing Concrete and Concrete Aggregates by Freezing and Thawing," Appendix to Report of Committee C-9 on Concrete and Concrete Aggregates. *Proceedings*, Am. Soc. Testing Mats., Vol. 32, Part I, p. 364 (1932).

1933

(15) L. O. Hanson, "Weathering Tests of Concrete," *Proceedings,* Highway Research Board, Vol. 13, Part I, p. 303 (1933).

(16) Inge Lyse and J. M. Holme, "Durability Studies of Concrete and Aggregates," *Proceedings,* Am. Concrete Inst., Vol. 30, pp. 121–128 (1933).

1934

(17) H. F. Gonnerman, "Study of Cement Composition in Relation to Strength, Length Changes, Resistance to Sulfate Waters and to Freezing and Thawing of Mortars and Concrete," *Proceedings,* Am. Soc. Testing Mats., Vol. 34, Part II, p. 244 (1934).

1935

(18) Inge Lyse, "Effect of Brand and Type of Cement on Strength and Durability of Concrete," *Journal,* Am. Concrete Inst., January–February, 1935; *Proceedings,* Vol. 31, p. 247.

(19) M. O. Withey, "Freezing and Thawing, Permeability and Strength Tests on Vibrated Concrete Cylinders of Low Cement Content," *Journal,* Am. Concrete Inst., May–June, 1935; *Proceedings,* Vol. 31, p. 528.

1936

(20) H. S. Mattimore, "Durability Tests of Certain Portland Cements," *Proceedings,* Highway Research Board, Vol. 16, p. 135 (1936).

1937

(21) H. H. Scofield, "Some Tests to Show Effect of Freezing on the Permeability, Strength, and Elasticity of Concretes and Mortars," *Proceedings,* Am. Soc. Testing Mats., Vol. 37, Part II, p. 306 (1937).

1938

(22) F. C. Lang, "Testing for Deleterious Materials in Concrete Aggregates," *Proceedings,* Highway Research Board, Vol. 18, Part I, p. 250 (1938).

(23) C. E. Wuerpel, "Factors Affecting the Testing of Concrete Aggregate Durability," *Proceedings,* Am. Soc. Testing Mats., Vol. 38, Part I, p. 327 (1938).

1939

(24) R. W. Carlson, "Remarks on Durability of Concrete," *Proceedings,* Am. Concrete Inst., Vol. 35, pp. 359–364 (1939).

(25) Bert Myers, "Effect of Variations in Method upon the Results of Freezing and Thawing Mortars," *Proceedings,* Am. Soc. Testing Mats., Vol. 39, p. 930 (1939).

(26) M. O. Withey, "Factors Affecting the Resistance to Freezing and Thawing of Vibrated Concrete Made of Crushed Dolomite," *Proceedings,* Am. Concrete Inst., Vol. 35, pp. 553–560 (1939).

1940

(27) F. V. Reagel, "Freezing and Thawing Tests of Concrete," *Proceedings,* Highway Research Board, Vol. 20, p. 587 (1940).

1941

(28) E. R. Dawley, "Correlation Between Rapid Immersion and Standard Four Cycle per Day Freezing and Thawing Tests," *Proceedings,* Am. Concrete Inst., Vol. 37, p. 157 (1941).

(29) C. A. Hughes and K. A. Andersen, "The Effect of Fine Aggregate on the Durability of Mortars," *Proceedings,* Am. Soc. Testing Mats., Vol. 41, p. 987 (1941).

1942

(30) Fred F. Bartel, "Effect of Aggregate Characteristics on Durability of Concrete," Thesis for degree Master of Science, University of Maryland, (1942).

(31) H. H. Munger, "The Influence of the Durability of Aggregate Upon the Durability of the Resulting Concrete," *Proceedings,* Am. Soc. Testing Mats., Vol. 42, pp. 787–803. Discussion, pp. 804–807 (1942).

(32) Stanton Walker and Fred F. Bartel, "Effect of Size of Specimen in Studies of Durability of Concrete as Affected by Aggregates," ASTM BULLETIN, No. 118, October, 1942, p. 37.

1943

(33) E. D. Axon, T. F. Willis and F. C. Reagel, "Effect of Air-Entrapping Portland Cement on the Resistance to Freezing and Thawing of Concrete Containing Inferior Coarse Aggregate," *Proceedings,* Am. Soc. Testing Mats., Vol. 43, p. 981 (1943).

1944

(34) H. F. Gonnerman, "Tests of Concretes Containing Air-Entraining Portland Cements or Air-Entraining Materials Added to Batch at Mixer," *Proceedings,* Am. Concrete Inst., Vol. 40, p. 477 (1944).

(35) Harman S. Meissner, "Laboratory Freezing and Thawing Tests on Concretes Containing Various Air-Entraining Agents," *Proceedings*, Am. Concrete Inst., Vol. 40, p. 517 (1944).

(36) Stanton Walker, "Resistance of Concrete to Freezing and Thawing as Affected by Aggregates," *Circular 26*, Nat. Sand and Gravel Assn.

(37) Stanton Walker, "Freezing and Thawing Tests of Concrete Made with Different Aggregates," *Journal*, Am. Concrete Inst., June 1944, *Proceedings*, Vol. 40, p. 573.

(38) M. O. Withey, "Progress Report, Committee on Durability of Concrete," *Proceedings*, Highway Research Board, Vol. 24, p. 174 (1944).

1945

(39) T. C. Powers, "A Working Hypothesis for Further Studies of Frost Resistance of Concrete," *Proceedings*, Am. Concrete Inst., Vol. 41, pp. 245–272 (1945).

(40) C. E. Wuerpel and H. K. Cook, "Automatic Accelerated Freezing and Thawing Apparatus for Concrete," *Proceedings*, Am. Soc. Testing Mats., Vol. 45, p. 813 (1945).

1946

(41) W. F. Kellerman, "Effect of Use of Blended Cements and Vinsol Resin-Treated Cements on Durability of Concrete," *Proceedings*, Am. Concrete Inst., Vol. 42, p. 681 (1946).

(42) Symposium on Freezing-and-Thawing Tests of Concrete, *Proceedings*, Am. Soc. Testing Mats., Vol. 46, pp. 1198–1251 (1946). Introduced by a paper by M. O. Withey (See Ref. **44**) and consisting of prepared discussions by Charles W. Allen, Charles E. Wuerpel, W. H. Price, Raymond E. Davis, A. T. Goldbeck, F. V. Reagel, Bailey Tremper, Rudolph C. Valore, Jr., Stanton Walker, F. O. Anderegg and Guy H. Larson.

(43) C. E. Wuerpel, "Discussion of Symposium on Freezing and Thawing Tests of Concrete," *Proceedings*, Am. Soc. Testing Mats., Vol. 46, p. 1210 (1946).

(44) M. O. Withey, "Considerations Involved in the Making of Freezing and Thawing Tests on Concrete," *Proceedings*, Am. Soc. Testing Mats., Vol. 46, p. 1198 (1946) (See also Ref. **42**.)

1947

(45) S. L. Bugg, "Effects of Air Entrainment on Durability Characteristics of Concrete Aggregates," *Proceedings*, Highway Research Board, Vol. 27, p. 156 (1947).

(46) T. C. Powers and T. L. Brownyard, "Studies of the Physical Properties of Hardened Portland Cement Paste," *Journal*, Am. Concrete Inst., Oct. 1946–April 1947; *Proceedings*, Vol. 43.

1948

(47) J. B. Blackburn, "Freeze and Thaw Durability of Air-Entrained Concrete Using Indiana Aggregates," *Proceedings*, Highway Research Board, Vol. 28, p. 171 (1948).

(48) R. E. Bollen, "Durability of Portland Cement Concrete Determined by Primary Directional Freezing and Uniform Thawing," *Proceedings*, Highway Research Board, Vol. 28, p. 218 (1948).

(49) H. S. Sweet, "Research on Concrete Durability as Affected by Coarse Aggregate," *Proceedings*, Am. Soc. Testing Mats., Vol. 48, pp. 988–1016 (1948).

(50) H. S. Sweet and K. B. Woods, "Evaluation of Aggregate Performance in Pavement Concrete," *Journal*, Am. Concrete Inst., Vol. 19, June 1948, pp. 1033–1090.

1949

(51) Fred F. Bartel, "Effect of Air Entrainment on Resistance to Freezing and Thawing of Concrete Made with Questionable Aggregates," *Circular 31*, Nat. Sand and Gravel Assn.

(52) Delmar L. Bloem, "Effects of Various Admixtures on Concrete," *Publication 27*, Nat. Ready Mixed Concrete Assn.

(53) Delmar L. Bloem, "Additional Tests of Concrete Admixtures (Series 119)," *Publication 32*, Nat. Ready Mixed Concrete Assn.

(54) Stanton Walker and Delmar L. Bloem, "Effect of Soft Sandstone in Coarse Aggregates on Properties of Concrete," Special Publication of the Nat. Sand and Gravel Assn.

(55) Stanton Walker and Delmar L. Bloem, "Study of Sands in Concrete Subjected to Freezing and Thawing," *Circular 34*, Nat. Sand and Gravel Assn.

1950

(56) Stanton Walker and Delmar L. Bloem, "The Problem of Deleterious Particles in Aggregates," *Circular 35*, Nat. Sand and Gravel Assn.

(57) Stanton Walker and Delmar L. Bloem, "Tests of Admixtures for Concrete," *Publi-*

cation 28, Nat. Ready Mixed Concrete Assn.

1951

(58) H. S. Sweet, "Evaluation of Mortar Degree of Saturation as an Influence in Concrete Freezing and Thawing Tests," paper presented at 30th Annual Meeting of Highway Research Board, January 1951, for inclusion in *Proceedings,* Highway Research Board, Vol. 30.

DISCUSSION

MR. BRYANT MATHER.[1]—The authors have made a very useful and valuable contribution to the difficult question of how to test concrete by freezing and thawing, both by having developed so efficient and versatile an apparatus and by having described its operation and performance so well. I agree with most of their opinions and interpretations, as might be expected since I have been connected with the development and use of automatic apparatus of similar type by the Corps of Engineers for some time.

I doubt, however, that they mean, as they seem to state, that the standardization of procedure for freezing-and-thawing tests is not desirable. I hope that they feel, as I do, that, *for certain purposes,* a rigidly standardized freezing-and-thawing procedure is essential, but for other purposes variations in procedure may and should be employed for obtaining information that such variations can give.

I should like to suggest, by way of example, that for use in connection with such matters as ASTM methods for testing air-entraining additions and admixtures, a rigidly standardized and simplified freezing-and-thawing procedure is at the present time to be desired. The adoption of such a simplified standardized procedure, however, should not be interpreted as an attempt to suppress research and development work on other procedures, or suppress the fruitful field of studies of the effect of changes in procedure.

I should also like to commend the authors for the restraint they have exercised in concluding that their results indicate only that freezing in water is more severe than freezing in air and do not afford evidence that one is more significant than the other. I hope that the equipment described will be used to develop data on this point, since such equipment is unusually well adapted for such studies.

I hope that they will investigate the relative reproducibility of tests made by freezing in air and freezing in water. In his discussion several years ago, Mr. Walker states, "It seems reasonable that much of the disagreement in tests results of supposedly identical specimens, identically treated, may be due to uncontrolled (perhaps uncontrollable) differences in the amount of moisture taken up during the thawing operation."[2] It has seemed to me that this factor constitutes a strong argument in favor of freezing in water, since it would seem reasonable to assume that the problem of the take-up of moisture during thawing can be eliminated entirely when the specimens are frozen in water.

Mr. Walker has emphasized that the objective is to produce an exposure sufficiently severe to distinguish among concretes of different qualities. At present, the decision as to whether the distinguishing among concretes that is done by a specific test, is actually proportional to quality, depends on one's subjective judgment of quality or upon one's evaluation of similar concrete in use. From data presented in the paper, it is clear that freezing in water (particularly with reference to the cooperative work with the Corps of Engineers) indicates greater difference in quality between the gravel concrete and the trap rock concrete. Depending upon one's judgment of the relative quality of those two concretes, either freezer No. 1 or freezer No. 2 is giving the right answer. It is evident that if the objective of the tests had merely been to get a relative indication of which concrete was more resistant, without regard to the magnitude of the differences, the freezing in water would produce wider differences and, therefore, more clear-cut differentiation.

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[2] Discussion prepared by Stanton Walker of Symposium on Freezing-and-Thawing Tests of Concrete, *Proceedings,* Am. Soc. Testing Mats., Vol. 46, p. 1240 (1946).

The last point that I should like to make concerns the matter of the apparent anomaly between weight loss and dynamic modulus loss in the trap rock concrete tested by the Corps of Engineers. The authors state, "The trap rock concrete showed greater loss in weight and lesser loss in dynamic modulus of elasticity as compared with the quartz gravel concrete." I feel that this comparison should include consideration of the fact that the water-cement ratio of the gravel concrete was 0.84 gal per bag lower than that for the trap rock concrete. It seems to me likely that the lower water-cement ratio of the cement paste in the gravel concrete made that paste more resistant to freezing and thawing than was the cement paste of higher water-cement ratio in the trap rock concrete. This would tend to explain the greater scaling of the trap rock concrete. On this basis, I suggest that the actual difference in durability is greater than the results indicate, since the trap rock concrete had to overcome the handicap of a less durable higher water-cement ratio paste.

In summary, I feel that, for certain purposes, therefore, the lack of standardization of freezing-and-thawing test procedures is not as it should be; that freezing in water has certain advantages over freezing in air; and that the apparently anomalous relation between weight loss and dynamic modulus of elasticity results in tests reported for gravel and trap rock concrete probably reflects differences in water-cement ratio.

MR. STANTON WALKER (*author*).—I wish to thank Mr. Mather for his very excellent discussion of our paper. He has implied a lot of questions to which I do not know the answers. However, I must say that we do mean it when we say that we feel that the time has not yet arrived for determining upon a standardized test procedure. Enough information is not yet available on the significance of the several approaches.

That is not to say that there is no work to be done in the field of standardization. We now have a large number of methods, the details of which depend greatly upon equipment available, which fall into a few broad categories. A very useful purpose can be served by standardizing within these several categories and thus reducing an uncoordinated miscellany of approaches to a few orderly approaches covering about the same range.

The Subcommittee of Committee C-9 dealing with methods of testing concrete for resistance to freezing and thawing, of which H. K. Cook of the Corps of Engineers is chairman, has a very useful purpose to serve if it will prepare descriptions of a small, but sufficient, number of procedures to cover the range now encompassed by the many approaches. On the other hand, I feel that committee will be striving for an unattainable objective if it endeavors to standardize on a single test method with the expectation that it will find general acceptance.

Mr. Mather indicates that he does not

believe that such standardization would suppress research. I feel that it is unavoidable that it would do just that. If an organization so authoritative as the American Society for Testing Materials says "here is the standard way to make freezing-and-thawing tests," a very large majority of those who make such tests will follow that standard.

With respect to Mr. Mather's point concerning the significance of the difference in water ratio between the two classes of concrete tested in both our equipment and that of the Corps of Engineers, I recognize that the differences in results may, perhaps, be accounted for by difference in water ratio. Nevertheless, whatever the reason, these two methods of measuring effects of freezing and thawing did give conflicting indications, and I think we need to iron out the significance of the many factors of this kind before we proceed much further toward standardization.

MR. C. F. MOHR.[3]—I note that in test results put out by the Corps of Engineers, the complete physical properties of the aggregate are given. Everybody knows there is a wide difference if one is testing crushed stone against gravel, or gravel against trap rock; and being in the trap rock business, I know of about a dozen different trap rocks that have different physical properties. That would apply to all aggregates, and I think if all organizations would follow the practice of the Corps of Engineers, in giving the complete physical properties of the aggregates the results would be of more value to an engineer who is trying to find out just what these tests represent.

The U. S. Corps of Engineers, Waterways Experiment Station, Vicksburg, Miss., made tests somewhat similar to those by Walker and Bloem. The Corps of Engineers gives complete data on physical properties of aggregates and values of all other factors that influence final results in Corps of Engineers *Bulletins Nos. 30 and 34* and *Technical Memorandum No. 6–307.*

I believe it is meaningless to publish results of tests where all the physical properties of aggregates are not mentioned.

MR. D. L. BLOEM (*author*).—There can be no doubt that all available data concerning aggregate characteristics should be furnished when comparisons are being made to evaluate aggregate performance. In this paper, however, it was the performance of a particular piece of testing apparatus which was under scrutiny and not the relative merits of various aggregates. In the interest of presenting as much data as possible pertinent to the subject, in a limited space, only the minimum information considered necessary to demonstrate the probable relative quality of the various concretes was given.

MR. R. R. LITEHISER.[4]—I should like to ask a question about one of the practical aspects that results from this testing. The authors show improvement in resistance to freezing and thawing that accompanied certain modifications in processing. Many of us have aggregates we would like to improve and are interested in these remedial processing methods.

MR. BLOEM.—Figures 5 and 6 deal with aggregate processing. Figure 5 shows a comparison between an untreated gravel and the same gravel after processing in heavy liquid to remove lightweight particles. In Fig. 6, the comparison is between natural gravel graded from 1 in. down and gravel from the same source identically graded but secured by crushing 1 to 2-in. material.

MR. EDWIN P. PITMAN.[5]—I should like to ask whether there is any correlation between the number of cycles per week, whether it be the old-fashioned way where we ran one cycle per day or the new method where 50 cycles are run per day. We use a similar test on ceramics. In the Lincoln Tunnel, we had wall tile on which we ran tests successfully for years. Later we used ceramic lane markers 3 in. thick. The Specification Division used the same autoclave test, raising the pressure to 100 psi in 1 hr. The lane markers failed. We then changed the rate of heat transfer (time to reach the pressure of 100 psi) to compensate for the thicknesses of the two materials and the lane markers withstood the test.

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[3] Sales Engineer, Kingston Trap Rock Co., Kingston, N. J.

[4] Chief Engineer, Testing and Research Lab., Department of Highways, Columbus, Ohio.

[5] Engineer of Inspection, The Port of New York Authority, Engineering Dept., New York, N. Y.

Do you have anything on the effect of thermal shock caused by the number of cycles per day, whether one or 50 cycles?

MR. BLOEM.—At the present time, we have no direct comparisons between our old apparatus which operates at the rate of 1 cycle per day and our new one which operates at the rate of about 8 cycles per day. We are at present making such comparisons. However, as regards thermal shock, I should like to point out that the actual rate of freezing and thawing in the 1-cycle-a-day apparatus is essentially the same as in the automatic machine. The only difference is that, in the former, the specimens are brought down to freezing, maintained there for a considerable period of time, then brought up to thawing and maintained at the higher temperature. The difference in number of cycles per day lies in the length of time the maximum and minimum temperatures are maintained rather than in the time required for those temperatures to be reached.

MR. T. F. WILLIS[6] (*by letter*).—Messrs. Walker and Bloem have described a mechanically excellent apparatus for obtaining a rapid laboratory freezing-and-thawing cycle on concrete with a minimum of labor. That they and other concrete technologists should strive for this goal is in accord with the tradition of our great country. The United States is famous as the land of volume production; more automobiles, more airplanes, more this and that produced per time and labor unit than in any other country on the globe. Now it seems from this and other recent papers that we intend to obtain more laboratory freezing-and-thawing cycles per day than anyone else. In the accomplishment I see a distinct possibility that our reward may be notoriety rather than fame.

The primary purpose of a laboratory test of a structural material is to evaluate some property which is necessary for the material to resist forces engendered under service conditions. The secondary purpose is to accomplish this in a more economical and less time-consuming manner than would be possible by empirical trials under actual service. In consummating this last, care must be taken to see that the methods of acceleration do not materially change the magnitude of the forces, resistance to which is to be evaluated. There is a definite possibility that neither of the freezing tests described in this paper conforms to this requirement.

Numerous bits of evidence and T. C. Powers' exposition of the hydraulic pressure hypothesis indicate that rate of cooling is a prominent factor in determining the magnitude of the stress in concrete resulting from frost action; in fact, is directly proportional thereto under some conditions. Consider a concrete proposed for use in pavement. According to a 12-year record of the Missouri State Highway Department and reports of other agencies, the fastest rate of cooling to which pavements in this country are subjected under service conditions is 6 F per hour; and probably 75 per cent of the time the rate does not exceed 4 F per hour. One of the test procedures described in the paper uses a rate of cooling of about 6 times and the other 15 times that to be expected under natural weathering. If rate of cooling is a factor in the deterioration of concrete exposed to frost action, the acceleration achieved by these test procedures is somewhat analagous to accelerating a test of a glass shelf, intended as a support for a light flower pot, by hitting it with a sledge-hammer. It would appear that any attempt to predict the frost resistance of pavement concrete under service conditions, and particularly the relative frost resistance of different concretes, from results of such test procedures is fraught with danger.

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[6] Chief, Research Division, Bureau of Materials, Missouri State Highway Department, Jefferson City, Mo.

Probably Messrs. Walker and Bloem and the Engineers of the Waterways Experiment Station are fully cognizant of this danger. In interpreting their results they may have means of eliminating it. However, many others who are called on to make and interpret freezing tests of concrete will not have so wide a knowledge. It is to these latter that these cautionary remarks are addressed.

MR. STANTON WALKER (*author*).—Mr. Willis has raised a question which concerns all of us—namely, that of the significance of freezing-and-thawing tests and, particularly, of the greatly accelerated ones which have come along with the development of automatic apparatus. It is a good question. Much investigation is required before anything approximating a complete answer can be given.

It should be pointed out that increasing the number of cycles per day, which is made practicable by the automatic machines, does not necessarily increase the severity of the cycle. The automatic machine, described in our paper as producing 8 cycles each 24 hr, freezes and thaws at about the same rate as our manually operated machine which produces only 1 cycle each 24 hr. In the earlier machine the specimens merely remained frozen, or thawed, until the arbitrary time intervals of 7 or 17 hr, fixed by the conventional work day, had expired. It would appear that the new machine may do no more than get substantially the same answer as the old machine, but more quickly. This reasoning is supported by only a meager amount of data and additional information is being secured.

As to the significance of freezing-and-thawing tests *per se*, I am sure no one would attempt to translate numbers of cycles in the laboratory to years of service. However, I believe that there is evidence to indicate that suitable procedures rate the durability of different concretes, in relation to each other, in a significant manner. At least, I am confident that a variety of procedures can be used which will indicate one combination of materials as being likely to give better service than another.

The Missouri State Highway Department, with which Mr. Willis is associated as Research Engineer, was one of the pioneers in investigating freezing-and-thawing test methods and in arriving at a procedure which seemed to give results for different aggregates consistent with experiences in actual service.[7] That procedure provided for the use of saturated aggregates in concrete frozen in air at about 0 F and thawed in water at approximately 40 F. Since 1940 we have been using substantially that cycle in our laboratory and the new automatic machine is designed to continue it.

Our tests, since 1940, have included a large number of aggregates which have been used for many years. While no quantitative comparisons in terms of length of service can be made, the evidence is convincing that a reasonably good job can be done in arranging such aggregates in the order of their durability in concrete.

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[7] F. V. Reagel, "Freezing and Thawing Tests of Concrete," *Proceedings*, Highway Research Board, Vol. 20, p. 587 (1940).

Mr. Willis has pointed to the dangers of materially changing the "magnitude of the forces,"—that is, in the testing procedure as compared with nature. That is a real danger. Undoubtedly, the rate of freezing and thawing is a pertinent factor. However, it is believed that "thermal shock" is an even more important one—and of much more importance in some procedures than in others.

Consider freezing in air and thawing in water. The transition from a thawed condition to a frozen one, in air, involves a minimum of "shock." The "shock" occurs in the transition from a frozen condition to a thawed one when the 40 F water comes in direct contact with the 0 F specimen. The specimen is in a contracted condition and it may be reasoned that the principal stress induced will be compressive, which concrete is well equipped to withstand. Hence, such "shock" is not unduly destructive.

On the other hand, when specimens are frozen and thawed in water by the circulation of brine about tightly fitting metal cans containing them, there is "shock" in both the heating and cooling cycles and, in the case of the latter, with the specimen being in an expanded condition, the principal stresses induced are tensile, which concrete is poorly equipped to withstand. My observations suggest that test methods involving "thermal shock" in the cooling cycle may cause the thermal characteristics of the aggregates to have a greater influence on the results than they would have under service conditions.

SOME PROPERTIES AFFECTING THE ABRASION RESISTANCE OF AIR-ENTRAINED CONCRETE*

By L. P. Witte and J. E. Backstrom[1]

Synopsis

Sixty-six concrete mixes covering a range of eleven water-cement ratios varying from 0.40 to 0.70 by weight and six air contents varying from 0.2 to 16.8 per cent were included to determine the effect of entrained air and other properties on the abrasion resistance of concrete. Results of the shotblast test, which was used for comparing the abrasion resistance of the concrete in this investigation, are correlated with the erosion obtained under exposure to the forces of the cavitation in water flowing at high velocities.

In recent years the Bureau of Reclamation has specified that all concrete contain purposely entrained air because of the advantages gained in workability of the fresh concrete and durability of the hardened concrete. Because in many instances structures built by the Bureau are subjected to the abrasive action of sands carried in flowing water and to other forms of abrasion, it was deemed advisable to ascertain what effect entrained air would have on the abrasion resistance of the concrete.

Many tests (**1, 2, 3, 4, 5**)[2] made on non-air-entrained concrete have shown that the abrasion resistance of concrete is influenced more by the strength of the concrete than by any other property. Operations and procedures such as proper curing, removal of water by the vacuum process, low slump, and low water-cement ratio, all of which tend to increase the strength of the concrete at the surface or throughout the mass, also increase the abrasion resistance of the concrete. In the case of air-entrained concrete, the strength is usually reduced as the percentage of air is increased, and it was the aim of the investigation described in this paper to determine whether the abrasion resistance decreased in proportion to this reduction in strength and whether concrete containing entrained air could be made as resistant to abrasion as non-air-entrained concrete even though it might have a lower density.

Since this investigation was made primarily to determine the effects of entrained air on the abrasion resistance of concrete, the effects of curing, finishing, placing, and handling procedures were not included. It is realized that the entrainment of air reduces bleeding of concrete. This may have a material effect on the abrasion resistance of slabs to the degree that bleeding may influence the water-cement ratio and strength at the top surface. However, it is believed that any effect on abrasion, because of this, may also be tied in with the strength of the concrete mass.

The specimens in this investigation were all cured in an identical fashion, and the abrasion determinations were made on the same specimens that were

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* Presented at the Fifty-fourth Annual Meeting of the Society, June 18–22, 1951.

[1] Materials Engineers, Bureau of Reclamation, U. S. Department of the Interior, Denver, Colo.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1153.

TABLE I.—CONCRETE MIX DATA.

Series AT (Abrasion Tests)

Sand grading, per cent	Pan	#100	#50	#30	#16	#8
	5	16	24	25	15	15

Cement 6,188
Specific gravity 3.11

Aggregate Platte
Specific gravity 2.59

ABS 0.89 per cent

Gravel grading, per cent	#4—⅜″	⅜″—¾″
	40	60

Date cast 1947	Mix No.	Parts of Aggregate, by weight	Total Parts, by weight	Per cent Sand	Net Water-Cement Ratio, by weight	Slump, in.	Unit Weight Fresh Concrete, lb per cu ft	Water Content, lb per cu yd	Cement Content, lb per cu yd.	Vinsol Resin Solids, gr per cu yd	Air Content, per cent		Unit Compressive Strength, 29-day				Abrasion loss, per cent by volume				Voids-Cement Ratio, V/C	Vinsol Resin Solids, per cent by weight of cement	Specific Gravity, 28-day unit weight, lb per cu ft
											Fresh concrete	28-day concrete	1	2	3	Avg	1	2	3	Avg			
4– 1	24B	3.34	4.74	43	0.40	2⅝	145.8	333	832	0.0	0.8	0.2	6150	6150	6200	6170	0.160	0.119	0.129	0.136	1.29	0.0	147.0
3–26	65	3.38	4.78	43	0.40	2⅞	141.0	319	797	100.7	4.2	3.3	5060	5100	5450	5200	0.152	0.152	0.162	0.155	1.51	0.000279	143.0
3–26	66	3.50	4.90	43	0.40	3	137.7	304	760	139.0	6.6	4.5	5020	5210	5210	5150	0.136	0.118	0.114	0.123	1.69	0.000402	141.0
3–31	67	3.54	4.94	43	0.40	2⅝	133.3	292	730	202.0	9.9	7.6	4070	4170	4280	4170	0.202	0.213	0.203	0.206	1.95	0.000610	136.4
3–31	68	3.70	5.10	43	0.40	2⅞	129.7	275	687	327.0	12.4	10.3	3470	3730	3610	3610	0.263	0.238	0.217	0.239	2.19	0.001050	132.7
4– 3	69	4.05	5.44	43	0.40	3	122.8	244	610	1,585.0	17.3	14.7	2560	2710	2600	2620	0.368	0.307	0.273	0.316	2.73	0.005730	126.6
2–13	22	3.77	5.20	43	0.43	2¾	146.2	327	760	0.0	0.4	0.0	5960	5960	6430	6120	0.187	0.163	0.150	0.167	1.36	0.0	148.3
2–24	25	4.00	5.43	43	0.43	3⅛	142.7	305	710	80.6	3.6	3.3	5110	4970	5020	5030	0.172	0.186	0.182	0.180	1.60	0.000250	143.1
2–24	26	4.17	5.60	43	0.43	2⅞	139.8	290	674	109.0	5.7	5.2	4700	4320	4460	4490	0.223	0.224	0.201	0.216	1.78	0.000357	140.3
2–26	27B	4.24	5.67	43	0.43	2½	133.1	273	635	166.0	9.8	7.2	3400	3660	3720	3590	0.320	0.326	0.256	0.301	2.14	0.000576	137.0
2–26	28B	4.49	5.92	43	0.43	2½	131.0	257	597	216.0	11.9	9.0	3520	3470	3290	3430	0.266	0.258	0.272	0.265	2.38	0.000797	135.4
4–23	70	4.72	6.15	43	0.43	2⅝	123.4	233	542	1,400.0	17.2	16.2	2560	2320	2510	2460	0.659	0.706	0.634	0.667	2.99	0.005689	125.0
2–13	21	4.08	5.54	43	0.46	3	145.7	327	710	0.0	0.6	0.0	5750	5660	5730	5710	0.174	0.177	0.174	0.175	1.46	0.0	148.2
2–24	29	4.32	5.78	43	0.46	3½	141.2	303	660	92.0	3.8	3.6	4470	4090	4170	4240	0.218	0.214	0.208	0.213	1.73	0.000308	141.6
2–24	30	4.51	5.97	43	0.46	3	139.0	289	628	109.0	5.9	6.2	3860	3680	3750	3760	0.229	0.222	0.212	0.221	1.92	0.000382	138.3
2–26	31	4.74	6.20	43	0.46	3¼	133.0	267	580	123.7	10.4	7.8	3500	3680	3310	3500	0.301	0.277	0.293	0.291	2.37	0.000470	136.9
4– 2	32A	4.97	6.43	43	0.46	2¾	129.6	250	545	252.0	12.6	10.8	2870	2710	2830	2800	0.311	0.312	0.233	0.285	2.65	0.001019	132.0
4– 7	71	5.45	6.91	43	0.46	3¼	119.4	215	467	1,015.0	20.0	17.7	1630	1790	1800	1740	0.575	0.511	0.462	0.516	3.66	0.004790	122.9
2–20	23A	4.67	6.16	43	0.49	2¾	146.8	316	645	0.0	0.0	0.0	4950	5200	5160	5100	0.201	0.201	0.211	0.205	1.56	0.0	149.2
2–27	33	4.90	6.39	43	0.49	2⅝	141.6	293	598	47.0	4.0	2.0	4320	4630	4600	4520	0.283	0.237	0.214	0.245	1.87	0.000173	144.7
4– 1	34A	5.03	6.52	43	0.49	3¼	138.4	280	573	82.0	6.5	4.7	3680	3710	3750	3710	0.229	0.228	0.221	0.226	2.12	0.000315	141.0
3– 3	35	5.12	6.61	43	0.49	3¼	132.6	266	542	170.0	10.4	8.6	3010	2940	2940	2960	0.377	0.336	0.338	0.350	2.53	0.000692	135.2
3– 3	36	5.40	6.89	43	0.49	2¾	130.5	251	512	229.0	11.9	10.7	2580	2510	2550	2550	0.386	0.375	0.400	0.387	2.74	0.000987	132.2
4– 7	72	6.04	7.53	43	0.49	2⅝	121.1	213	435	517.0	18.8	16.7	1590	1870	1870	1780	0.562	0.468	0.444	0.491	3.79	0.002620	124.1
2–19	20	5.25	6.77	43	0.52	3	146.4	308	584	0.0	0.4	0.0	3860	4950	4810	4540	0.196	0.206	0.195	0.199	1.67	0.0	148.0
3–13	37A	5.45	6.97	43	0.52	2¾	142.5	288	552	54.0	3.4	1.6	4040	4300	4240	4190	0.220	0.246	0.235	0.234	1.95	0.000216	145.3
3– 3	38	5.52	7.04	43	0.52	3	137.6	275	528	112.0	6.8	5.2	3430	3380	3130	3310	0.334	0.296	0.290	0.306	2.30	0.000468	139.8
3–10	39	5.72	7.24	43	0.52	3⅜	133.3	258	497	146.5	10.0	7.6	2740	2800	2710	2750	0.423	0.399	0.387	0.403	2.67	0.000650	136.9
3–10	40	6.03	7.55	43	0.52	2¾	130.4	243	467	199.0	12.2	9.8	2510	2420	2280	2400	0.515	0.505	0.524	0.515	2.98	0.000940	134.0
4– 8	73	6.61	8.13	43	0.52	2⅞	120.5	208	401	447.0	19.4	18.2	1200	1400	1410	1340	0.924	0.665	0.630	0.740	4.13	0.002460	122.2
2–20	19A	5.60	7.15	43	0.55	2¾	146.4	304	553	0.0	0.4	0.0	4460	4460	4530	4480	0.300	0.279	0.254	0.277	1.75	0.0	148.3
3–13	41A	5.92	7.47	43	0.55	2⅝	142.0	282	513	58.5	3.4	1.3	3760	3800	3880	3810	0.299	0.282	0.276	0.285	2.06	0.000251	145.3
3–10	42	6.13	7.68	43	0.55	2½	139.7	271	493	87.5	5.8	3.3	3400	3540	3260	3400	0.312	0.303	0.311	0.309	2.33	0.000392	143.3
3–10	43	6.20	7.75	43	0.55	3	132.5	254	462	148.0	10.5	8.2	2440	2550	2370	2450	0.432	0.450	0.470	0.450	2.90	0.000706	136.0
3–12	44	6.36	7.91	43	0.55	3¼	130.8	246	446	164.0	11.9	9.6	2120	2260	2190	2190	0.427	0.397	0.429	0.418	3.12	0.000810	134.2
4– 8	74	7.08	8.63	43	0.55	3	120.7	208	378	399.0	19.1	17.7	1270	1360	1380	1340	1.023	0.791	0.773	0.862	4.36	0.002320	122.8

2-19	18	5.98	7.56	43	0.58	2⅞	146.5	303	523	0.0	0.4	0.0	4280	4490	4240	4340	0.262	0.262	0.251	0.258	1.84	0.0	148.3
3-11	45	6.31	7.89	43	0.58	2¾	142.1	282	486	45.4	3.7	1.9	3500	3500	3400	3470	0.251	0.233	0.243	0.242	2.20	0.000206	145.0
3-11	46	6.37	7.95	43	0.58	3⅛	138.4	272	470	80.0	6.4	4.6	2940	2970	2580	2830	0.305	0.295	0.287	0.295	2.51	0.000362	141.2
3-13	47	6.62	8.20	43	0.58	3⅛	133.6	256	440	128.0	9.9	7.4	2500	2570	2400	2490	0.448	0.438	0.419	0.434	2.98	0.000641	137.1
3-13	48	6.84	8.42	43	0.58	3	129.7	242	417	165.0	12.4	10.6	1950	1880	1870	1900	0.509	0.546	0.521	0.525	3.36	0.000872	132.5
4- 8	75	7.62	9.20	43	0.58	3⅜	120.1	205	353	350.0	19.6	17.8	1080	1130	1130	1110	1.174	1.007	0.883	1.021	4.72	0.002180	122.8
2-19	16	6.49	8.10	43	0.61	2⅝	146.5	299	490	0.0	0.4	0.0	3750	3780	3820	3780	0.238	0.273	0.265	0.259	1.95	0.0	147.7
3-17	49	6.76	8.37	43	0.61	2⅞	142.0	280	458	50.0	3.9	2.2	3200	3250	3180	3210	0.295	0.277	0.270	0.281	2.34	0.000241	144.3
3-19	50A	7.02	8.63	43	0.61	2¾	139.4	266	436	55.0	6.0	4.9	3080	3110	2870	3020	0.305	0.330	0.296	0.310	2.61	0.000278	141.0
3-24	51A	7.04	8.65	43	0.61	3⅜	133.0	253	415	94.0	10.2	8.1	2190	2300	2330	2270	0.513	0.457	0.442	0.470	3.18	0.000637	136.0
3-19	52	7.58	9.15	43	0.61	3⅜	130.2	234	384	102.0	12.1	11.3	1560	1950	1700	1740	0.557	0.505	0.518	0.527	3.55	0.000585	131.4
4- 8	76	8.15	9.76	43	0.61	3	120.6	204	334	282.0	19.4	18.0	970	1020	1040	1010	1.100	0.893	0.884	0.959	4.95	0.001860	122.5
2-19	15	6.93	8.57	43	0.64	2⅝	147.2	297	464	0.0	0.0	0.0	3380	3520	3540	3480	0.248	0.251	0.241	0.246	1.99	0.0	148.3
3-20	53	7.16	8.80	43	0.64	3⅛	141.5	278	435	40.0	4.0	2.4	3040	3060	3100	3070	0.338	0.321	0.295	0.318	2.47	0.000202	144.0
3-20	54	7.34	8.98	43	0.64	3⅛	138.2	266	415	55.3	6.6	4.7	2490	2620	2620	2580	0.372	0.355	0.331	0.352	2.82	0.000294	141.0
3-20	55	7.63	9.27	43	0.64	3⅜	133.0	248	387	85.5	10.3	7.8	1840	1910	1840	1860	0.502	0.532	0.487	0.507	3.40	0.000486	136.8
3-20	56	7.93	9.57	43	0.64	3½	130.0	235	367	101.0	12.3	9.7	1560	1640	1630	1610	0.596	0.554	0.480	0.543	3.75	0.000607	133.7
4- 9	77	8.82	10.46	43	0.64	3½	122.2	202	316	205.0	17.7	16.0	1110	1170	960	1080	0.805	0.870	1.020	0.898	4.92	0.001430	124.7
2-20	14	7.24	8.91	43	0.67	2⅝	146.5	299	446	0.0	0.0	0.3	3340	3360	3340	3350	0.349	0.342	0.336	0.343	2.09	0.0	148.3
3-24	57	7.56	9.23	43	0.67	3⅛	141.0	277	413	46.5	4.0	1.5	2830	2830	2760	2810	0.359	0.363	0.328	0.350	2.59	0.000248	144.7
3-24	58	7.79	9.46	43	0.67	2⅝	137.8	264	394	65.5	6.3	4.2	2390	2490	2300	2390	0.380	0.425	0.395	0.400	2.92	0.000366	141.0
3-24	59	8.20	9.87	43	0.67	3⅛	133.3	244	365	76.8	9.9	8.3	1700	1840	1520	1690	0.484	0.584	0.551	0.539	3.50	0.000464	135.4
3-25	60	8.50	10.17	43	0.67	3	130.6	233	347	87.1	11.8	10.4	1560	1660	1610	1610	0.597	0.556	0.534	0.562	3.93	0.000554	132.9
4- 9	78	9.56	11.23	43	0.67	3⅛	123.6	199	297	164.0	17.7	15.7	830	1010	920	920	1.185	1.061	0.965	1.071	5.21	0.001217	126.0
4- 1	13B	7.69	9.39	43	0.70	2½	144.7	291	416	0.0	1.2	0.0	3150	3180	3180	3170	0.296	0.282	0.275	0.284	2.33	0.0	146.5
3-25	61	8.24	9.94	43	0.70	2½	141.4	270	385	32.6	3.8	2.5	2580	2510	2440	2510	0.341	0.343	0.328	0.337	2.67	0.000187	143.5
3-25	62	8.38	10.08	43	0.70	3⅛	139.0	261	373	48.0	6.0	5.0	2260	2410	2330	2330	0.372	0.312	0.297	0.327	3.03	0.000284	140.7
3-26	63	8.82	10.52	43	0.70	3	133.5	240	343	70.0	10.1	9.2	1930	1810	1670	1800	0.412	0.379	0.421	0.404	3.88	0.000450	134.5
3-26	64	9.22	10.92	43	0.70	3⅜	130.3	226	322	86.3	12.3	11.0	1450	1200	1460	1370	0.519	0.543	0.464	0.508	4.18	0.000590	132.2
4- 9	79	10.20	11.90	43	0.70	3	122.3	194	278	145.0	17.5	15.5	710	900	830	810	1.263	1.093	1.093	1.149	5.47	0.001145	126.5
4-28	80	2.86	4.23	43	0.37	2+	145.8	344	930	0.0	0.9	1.4	7400	7610	7570	7530	0.114	0.125	0.108	0.116	1.20	0.0	145.0
4-28	81	2.19	3.53	43	0.34	2	145.0	377	1,110	0.0	0.7	1.2	7550	7760	7360	7560	0.102	0.091	0.094	0.096	1.09	0.0	144.5
5- 1	82[a]	3.13	4.53	46	0.40	2½	144.0	343	858	0.0	1.6	2.2	6840	6700	6730	6760	0.152	0.127	0.113	0.131	1.34	0.0	143.4
5- 1	83[a]	5.23	6.78	46	0.55	2¼	143.7	310	564	0.0	1.2	2.0	5050	4900	5060	5000	0.164	0.170	0.159	0.164	1.82	0.0	143.7
5- 1	84[a]	6.99	8.69	46	0.70	2½	143.7	313	447	0.0	2.0	1.1	2880	3220	3260	3120	0.286	0.296	0.272	0.285	2.42	0.0	144.0
5- 5	80[b]	2.50	3.90	100	0.40	0	137.8	382	955	0.0	3.5	2.5	6720	6700	6670	6700	0.166	0.169	0.170	0.168	1.44	0.0	140.4
5- 5	86[b]	3.72	5.27	100	0.55	0	135.0	381	692	0.0	4.8	2.9	4560	4600	4620	4590	0.275	0.253	0.229	0.252	2.08	0.0	137.9
5- 5	87[b]	4.98	6.68	100	0.70	0	134.4	380	544	0.0	5.0	3.3	3220	3130	3150	3170	0.326	0.319	0.305	0.317	2.66	0.0	136.7

[a] ⅜-in. maximum size aggregate.

[b] No. 4 maximum size aggregate

used in measuring air content and compressive strength. This was possible because it was found that within the limit of the tests the loss of abraded material had no effect on the compressive strength of specimens. The shotblast test was used for the abrasion determinations. A search of the literature and of previous tests made in this laboratory indicated that there was a correlation between all types of abrasion tests if they were properly made. Therefore, the shotblast test, being easy to make and easy to control, was selected.

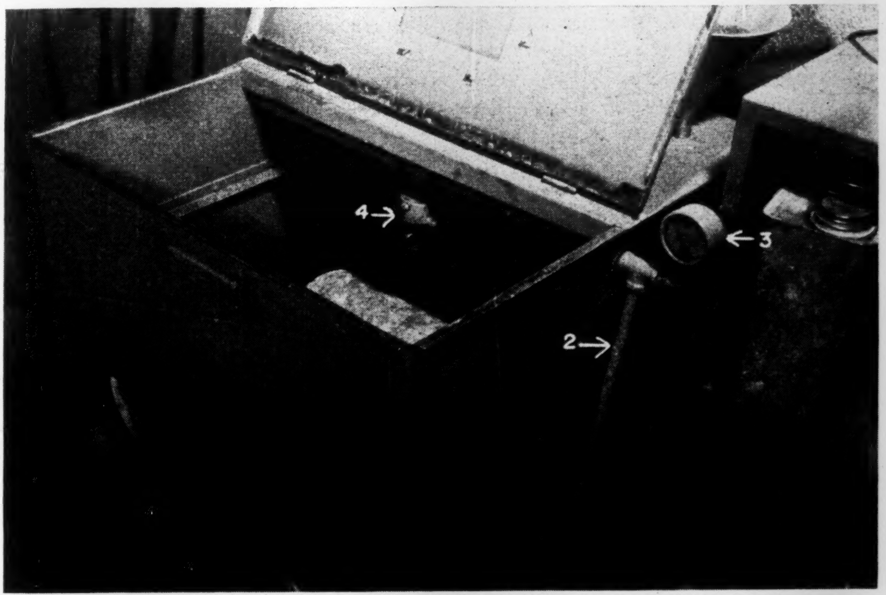
LABORATORY TESTS

Sixty-six concrete mixes, using $\frac{3}{4}$-in.-maximum size aggregate, were made to include six mixes for each of eleven water-cement ratios varying in increments of 0.03 from 0.40 to 0.70, inclusive. Air contents of fresh concretes averaged 0.2, 3.5, 6.6, 10.2, 12.1, and 16.8 per cent in the specimens representing each water-cement ratio. A tolerance of ±0.5 per cent was allowed on all air contents except the 16.8 per cent where 1.5 per cent was permitted, and where the batch did not meet this tolerance it was discarded. A 5 per cent solution of Vinsol resin was used as the air-entraining agent. Slump was maintained at 3 in. $\pm\frac{1}{2}$ in. in all mixes. Air content of the fresh concrete was gravimetrically determined on each specimen immediately after fabrication; however, the air contents calculated from the specific gravity of the cylinders after 28 days' fog curing were used in the comparisons of abrasive resistance.

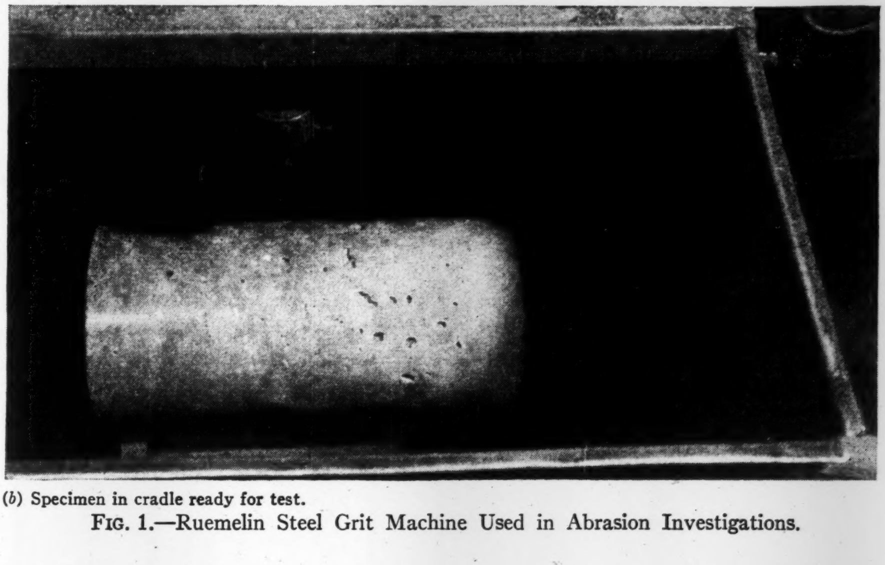
In order to extend the range of water-cement ratios and to investigate the influence of maximum size of aggregate beyond the limits of the main series, eight additional "plain" mixes were added to the program. These included two mixes of $\frac{3}{4}$-in. maximum size aggregate having water-cement ratios of 0.34 and 0.37, three mixes containing No. 4, and three mixes containing $\frac{3}{8}$-in. maximum size aggregate. For the latter two groups of mixes, water-cement ratios of 0.40, 0.55, and 0.70 were used, and represented the low, mean, and high ratios of the main series. All pertinent data concerning the properties of the concrete are shown in Table I. Concrete was mixed dry in a $\frac{3}{4}$-cu-ft pan-type mixer for 30 sec followed by 3 minutes' mixing after adding the water. Compaction of the concrete in the molds consisted of 20 seconds' vibration in each of two lifts with a 1-in. electric vibrator. Three 6 by 12-in. cylinders were fabricated from each mix, and were cured 28 days at 70 F, 100 per cent relative humidity. At the end of the curing period, specific gravities of the specimens were determined. Specimens were then dried for 24 hr in a room controlled at 70 F, 50 per cent relative humidity. After the drying period, each cylinder was subjected to the abrasion test and immediately thereafter, capped, and broken in compression. The loss of concrete during the abrasion test had no appreciable effect on the compressive strength.

There being no ASTM standard or other recognized test for determining the abrasion resistance of concrete, the literature on this subject was reviewed to aid in the selection of a suitable test method. It was found that nearly as many different methods were employed as there were investigators. The various methods used included the rattler-type tests (**1**, **2**), rotating abrasive disks (**4**), wheels or castors revolving in a fixed orbit on test slabs (**6**, **8**), water jets with and without abrasives (**3**), and air jets containing abrasives (shotblast) (**5**, **7**). From the review of these methods, it became evident that no one test so far devised has adequately measured the abrasion resistance of concrete under all conditions.

After review of these test methods and consideration of the merits and deficiencies of each, the shotblast method was selected for this investigation. It was felt that in rattler-type tests impact played too important a rôle; abrasive disks tended to become smooth, and nonremoval of eroded material tended to hasten this process. The water-jet sand method and that which employed



(*a*) General view of Ruemelin machine showing (1) grit reservoir, (2) air line, (3) pressure gage, (4) air-grit mixing amber and nozzle. Air pressure regulator not shown.



(*b*) Specimen in cradle ready for test.

Fig. 1.—Ruemelin Steel Grit Machine Used in Abrasion Investigations.

wheels and castors revolving in a fixed orbit required an excessive length of time to complete, and during this time the test specimen would undergo changes brought about by the additional moist curing in the former or by the drying in the latter method. It was for these reasons that the above-mentioned methods were eliminated. In contrast, the shotblast method was shown to be a rapid test which provided close control over the amount and angularity of the abrasive used and, through action of the air jet, removed all eroded material. The rapidity with which a specimen could be tested made this method ideal for an extensive investigation involving many specimens. Also contributing to its selection were the results of other tests made in the Bureau's laboratory showing this test to have highly significant correlation with the resistance of concrete to forces of cavitation.

The shotblast test, performed in a Ruemelin abrasion machine, consisted essentially of placing the test specimens in a cradle so that the surface to be tested was 4 in. from a $\frac{1}{4}$-in. nozzle through which the abrasive material was projected by air at 100-psi pressure. A total of 16,000 g of No. 20 broken steel shot were projected against the perimeter of each 6 by 12-in. cylinder at eight diametrically opposed spots, each spot receiving 2000 g. Since continuous use of the abrasive medium, with the resulting loss of angularity, would influence the test results, 25 lb of shot were allotted to each group of 18 cylinders representing concrete of the same water-cement ratio. In this manner the angularity of the shot was maintained nearly constant for each water-cement ratio group. After each use, care was taken to remove all eroded concrete particles from the shot. The high velocity of the jet resulted in rapid wear of the nozzle, and to minimize the influence of this variable on the results, a new nozzle was used for the three cylinders fabricated from each concrete mix. Photographs of the apparatus are shown in Fig. 1.

Method of Computing Abrasion Loss:

Abrasion loss was determined by weighing the specimen before and after test. This weight loss was then converted to a volumetric basis because weight alone does not take into account the air entrained in the concrete. The per cent loss by volume was calculated by first determining the percentage weight loss as measured by the difference in weight of the specimen (at 29 days' age) before and after test. Since the percentage loss by weight and the per cent loss by volume are identical for a given specimen, no further calculation is necessary. The 28-day air content was determined by dividing the actual 28-day unit weight of the concrete by the theoretical unit weight corrected to eliminate all entrapped air. This 28-day air content and the volumetric abrasion loss were used in all calculations and comparisons made. It should be noted that all determinations, such as compressive strength, air content, and abrasion loss, were obtained from the same individual cylinders; thus, any variations in air contents were reflected in both abrasion loss and compressive strength. It should also be borne in mind that the per cent loss by volume is only of importance for comparison in this investigation. Had the specimens been of greater volume, the amount of eroded material would have remained the same, but the loss in per cent would have decreased. A pictorial representation of the abrasion losses sustained by typical concretes tested in this investigation is shown in Fig. 2.

DISCUSSION

The results of this investigation show that compressive strength is the most important factor controlling the abrasion resistance of concrete. Air content and other properties of concrete such as density, water-cement ratio, cement content, etc., influence this resistance only in so far as they affect compressive strength.

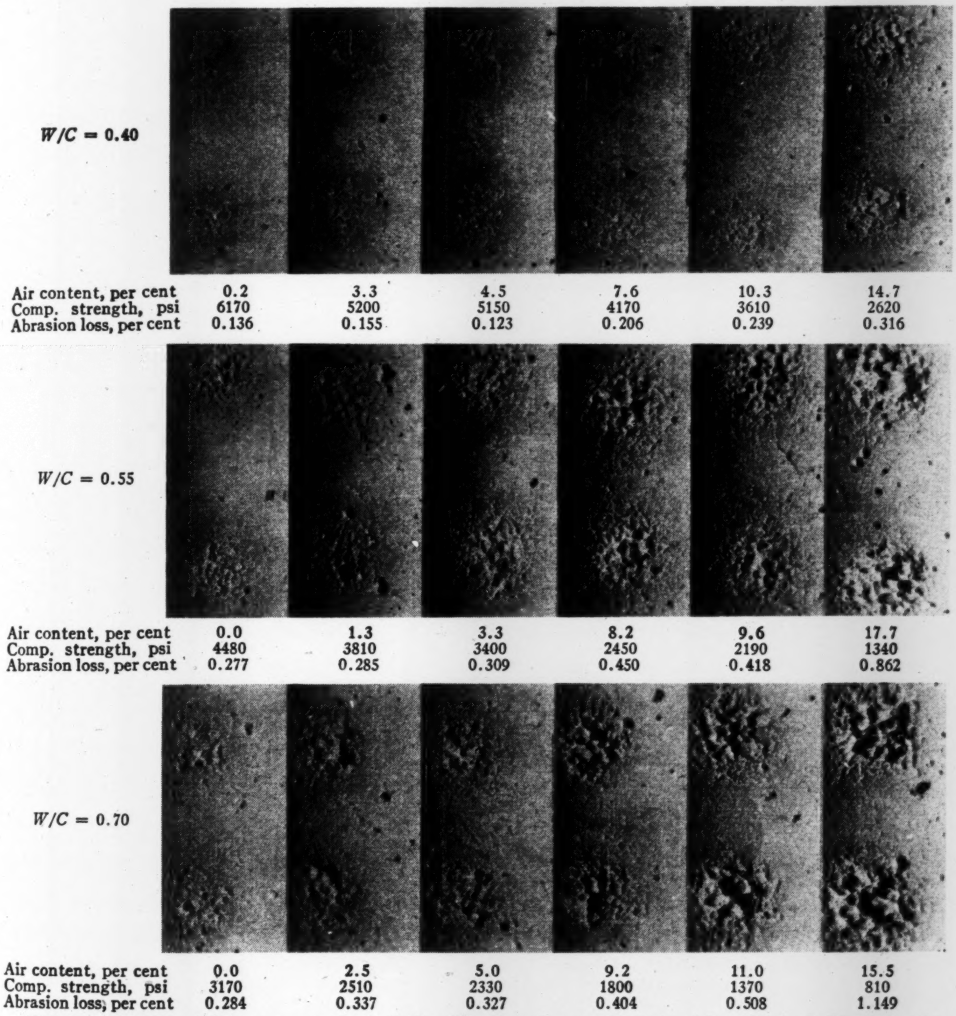

W/C = 0.40
Air content, per cent 0.2 3.3 4.5 7.6 10.3 14.7
Comp. strength, psi 6170 5200 5150 4170 3610 2620
Abrasion loss, per cent 0.136 0.155 0.123 0.206 0.239 0.316
W/C = 0.55
Air content, per cent 0.0 1.3 3.3 8.2 9.6 17.7
Comp. strength, psi 4480 3810 3400 2450 2190 1340
Abrasion loss, per cent 0.277 0.285 0.309 0.450 0.418 0.862
W/C = 0.70
Air content, per cent 0.0 2.5 5.0 9.2 11.0 15.5
Comp. strength, psi 3170 2510 2330 1800 1370 810
Abrasion loss, per cent 0.284 0.337 0.327 0.404 0.508 1.149

FIG. 2.—A Pictorial Representation of the Abrasion Losses Sustained by Typical Concretes in the Shot-Blast Test. Note that concretes of equal strengths have approximately equal abrasion resistance regardless of the amount of air entrained.

The influence of several of the properties of concrete on abrasion resistance is shown in Fig. 3 by contour curves in which density, compressive strength, and abrasion loss are represented. Points of equal abrasion loss are represented by the contour lines. It may be seen that although there is a general increase in strength as density increases, concretes having equal density cover a wide range of compressive strengths. For example, in concretes having a density of 140 lb per cu ft, the strengths range from 2400 to 4700 psi, and corresponding abrasion losses range from 0.4 to 0.2 per cent. In other words, the abrasion resistance (the reciprocal of abrasion loss) was increased 100 per cent by the increase in strength of 96 per cent. From these wide variations, it is concluded that density which, in effect, is but a reflection of the varying air contents in this series cannot be directly related to *abrasion* resistance. Density and air content, therefore, may be eliminated as indexes to the abrasion resistance of concrete.

Further examination of Fig. 3 shows that concretes of the same strength cover a wide range of densities. For example, strengths of 3500 psi may be obtained from concretes having densities from 132 to 150 lb per cu ft. However, it may be seen that the abrasion loss as represented by the contour line is relatively constant. This same trend is evident in all strength ranges and is the basis for the main conclusion drawn from this test series, that is, "that compressive strength is the most important factor controlling the abrasion resistance of concrete."

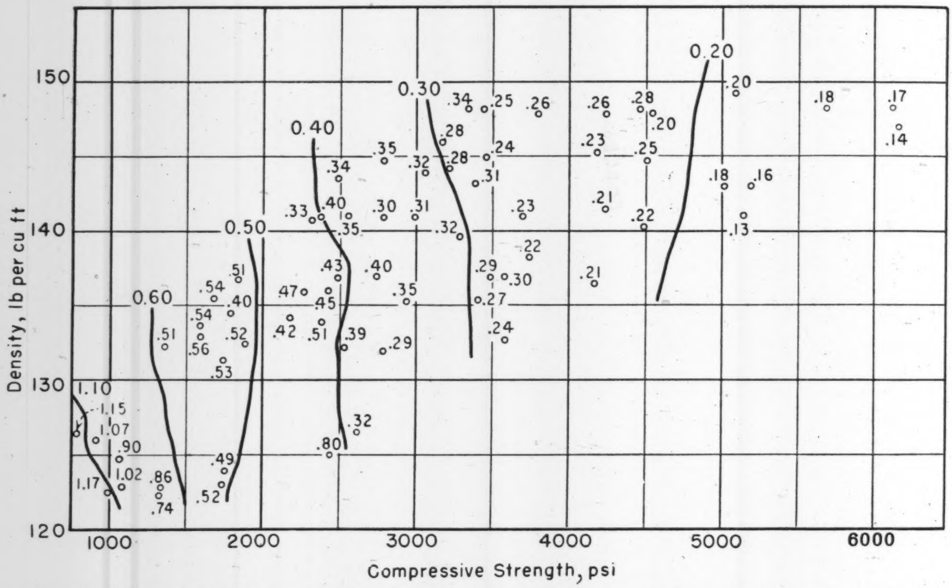


FIG. 3.—Compressive Strength Is the Most Important Factor Controlling the Abrasion Resistance of Concrete.

To demonstrate the close correlation existing between strength and abrasion loss, the concretes in the test series were grouped according to compressive strength. All of the $\frac{3}{4}$-in. maximum size aggregate mixes recorded in Table I are represented in the groups, each group including those concretes whose strength falls in the range S_n to $S_n + 0.10\ S_n$, where S_n is the compressive strength of concrete having the lowest compressive strength of the group. The first group (810 to 891 psi) so established includes the concrete having the lowest compressive strength in the test series (Mix No. 79, 810 psi). In the group only one concrete produced strength within this range. The second group (920 to 1012 psi) had as a base the next lowest strength in excess of the maximum limit, 891 psi, of the first group and included Mixes Nos. 78 and 76 which produced compressive strengths of 920 and 1010 psi, respectively. In this way a total of 16 groups was developed from the 68 mixes in the test series and each group includes from one to eight concrete mixes. The average compressive strength and average abrasion loss of the groups are compared in Fig. 4. In this figure it is shown that 800 psi concrete has virtually no resistance to abrasive action but as strength increases the resistance increases. In the strength range of average concretes, 3000 to 5000 psi, abrasion resistance is in direct proportion to strength and concrete of 3000

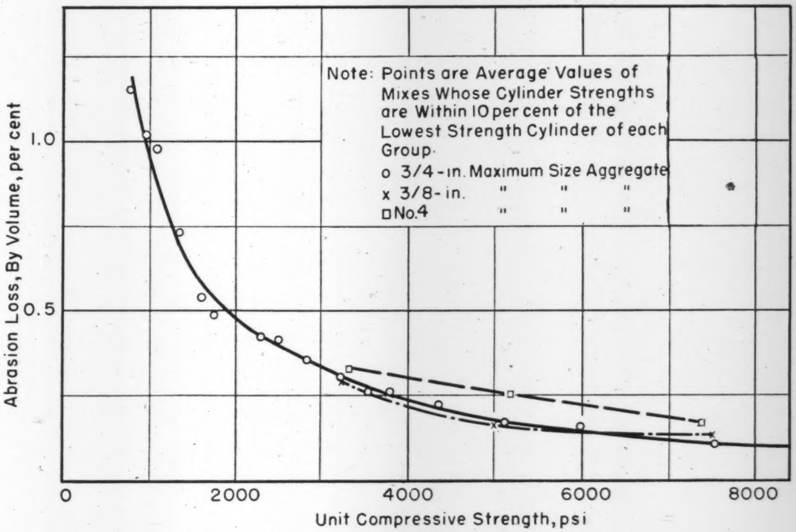


FIG. 4.—As Compressive Strength Increases Abrasion Loss Decreases.

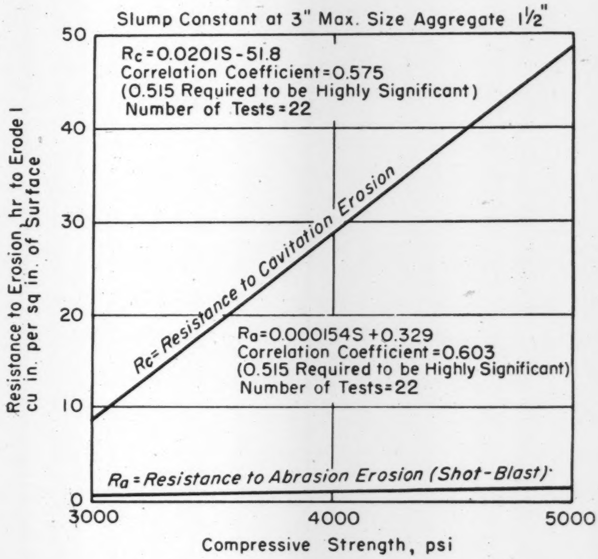


FIG. 5.—The Resistance of Concrete to Cavitation or Abrasion Erosion Varies Directly With Compressive Strength.

psi strength is only one-half as resistant to abrasion as 5000 psi concrete.

At first glance at the curve there appears to be little advantage to increasing the strength above 5000 psi; however, if abrasion resistance for 5000 psi concrete is considered to be unity, the abrasion resistance of 8000 psi concrete is 2, or twice as great as that of the 5000 psi concrete.

From the limited number of tests performed, results of which are also plotted in Fig. 4, the maximum size of aggregate appears to have little effect on the concrete's resistance to abrasion when compared on the basis of equal strength.

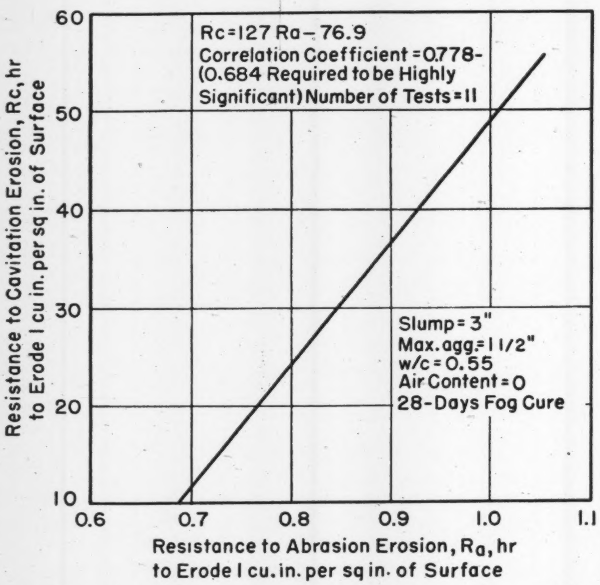


FIG. 6.—Abrasion Erosion Is a Reliable Index of Cavitation Erosion and Is Obtained in a Relatively Short Period of Time.

Further support of the importance of strength as an index to abrasion resistance is contained in Fig. 5. In this figure are plotted the strength-resistance to erosion relationships obtained from another extensive series of tests, whose data are not included in this paper, performed in the Bureau laboratories on concretes containing 1½-in. maximum size aggregate, representing strengths in the range of 3000 to 5000 psi. Resistance to erosion as shown on the curve is defined as the number of hours required to erode 1 cu in. per sq in. of surface exposed. Companion specimens in this series were subjected to shotblast erosion and erosion by cavitation. Briefly, the cavitation machine is of the Venturi type. Water flowing through this machine at sufficiently high velocity will result in the formation of vapor pockets in the low-pressure areas of the Venturi throat. These vapor pockets or bubbles are carried downstream until the pressure increase causes their collapse and it is this collapse which is extremely destructive to their boundary surfaces. Specimens were placed at this point of collapse.

The upper curve R_c, in Fig. 5 shows the relationship of compressive strength to this type of erosion, and it will be noted that an inordinately long test period is required in comparison to the shotblast method represented by the lower curve R_a. By examining the "abrasion erosion" curve R_a, representing the shotblast method, it is shown that results identical to those in the *main series*, Fig. 4, are obtained, concretes of 3000 psi having only one-half as much resistance to erosion (or abrasion) as 5000 psi concrete. Regardless of this time differential, a highly significant correlation, shown in Fig. 6, was obtained between the two types of tests. From this it may be said that the shotblast test furnishes an excellent index for predicting the relative erosion resistance of concrete. This relationship is expressed by the equation:

$$R_e = 127\ R_a - 76.9$$

In the foregoing discussion it has been shown that a very close relationship exists between compressive strength and abrasion loss. It has also been shown that density and air content, when considered independently, show no relationship. It is now proposed to discuss those recognized criteria for evaluating quality of concretes and their effect on abrasion resistance.

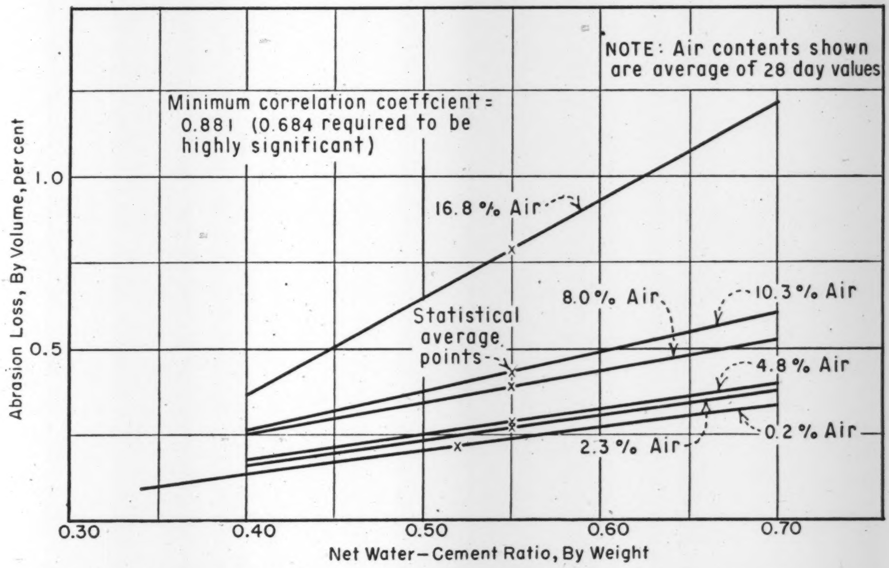


FIG. 7.—Abrasion Loss Is not Directly Dependent on Water-Cement Ratio or Air Content Alone.

Figure 7 shows the effect of water-cement ratio on abrasion loss for the six different air contents used in this investigation and also the effect of air content on abrasion loss for the eleven water-cement ratios used. Generally speaking, the curves are of the same trend and show an increase in abrasion loss caused by an increase in water-cement ratio. The effect of entrained air is reflected in the vertical displacement of the curves which shows an increase in abrasion loss accompanying an increase in air content for any given water-cement ratio. From this it may be seen that only with a constant air content will water-cement ratio have a definite influence on abrasion loss, the rates of abrasion loss varying according to the amount of air entrained. By means of the air plus water to cement (by volume) or voids-cement ratio, it is possible to combine the individual influences of water, cement, and air content, as they affect the abrasion loss or compressive strength. These factors vary widely in the 66 combinations of the main series. Each factor has its own influence on the properties of concrete, and these factors could be arranged in an infinite number of combinations. However, for any combination of these factors which produced the same voids-cement ratio relatively constant strengths and abrasion losses resulted. For example, the pertinent propertes

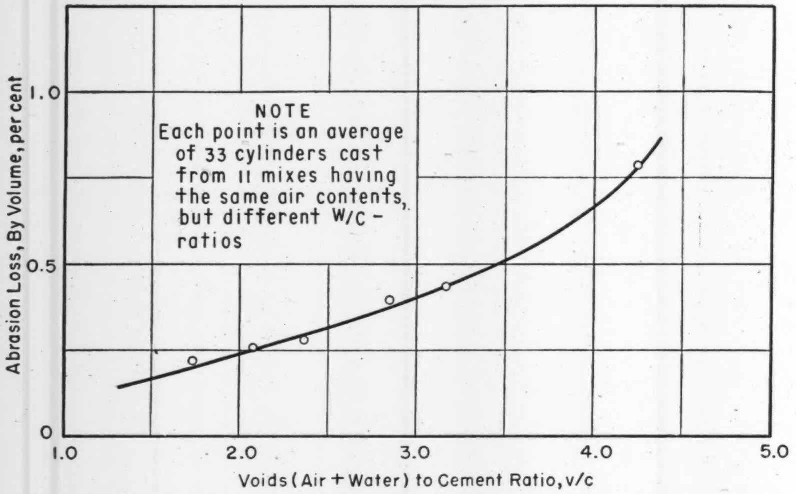


FIG. 8.—As the Voids-Cement Ratio Increases the Abrasion Loss Increases.

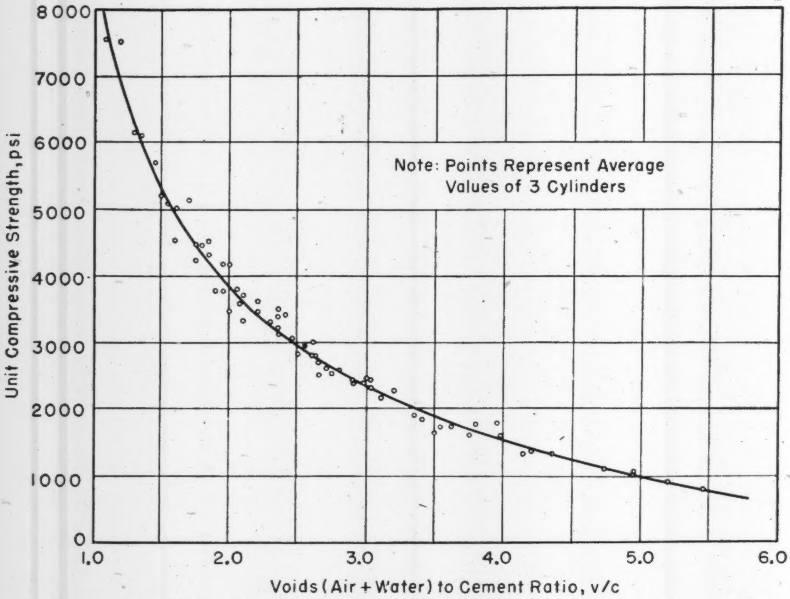


FIG. 9.—Compressive Strength is Dependent on the Voids-Cement Ratio Regardless of Water-Cement Ratio, Water, Cement or Air Contents.

of several mixes having voids-cement ratios equal to 2.70 ± 0.10 are tabulated below:

Water-Cement Ratio, by weight	Water Content, cu ft per cu yd	Cement Content, cu ft per cu yd	Air Content, cu ft per cu yd	Voids-Cement Ratio *V/C*, by volume	Compressive Strength, psi	Abrasion Loss, per cent
0.40	3.9	3.1	4.0	2.73	2,620	0.32
0.46	4.0	2.8	2.9	2.65	2,800	0.29
0.49	4.0	2.6	2.9	2.74	2,550	0.39
0.52	4.1	2.5	2.1	2.67	2,750	0.40
0.61	4.3	2.2	1.2	2.61	3,020	0.31
0.70	4.3	2.0	0.7	2.67	2,510	0.34

In order to show the close relationship existing between voids-cement ratio and both abrasion loss and compressive strength for the main series, Figs. 8 and 9 were prepared. These figures (together with Fig. 4) are included to show that strength is dependent on voids-cement ratio and that either strength or voids-cement ratio is a good index to abrasion loss.

From a practical standpoint, and for a given set of materials and conditions voids-cement ratio is as good an index of abrasion loss as is strength. However, since strength is a universally accepted measure of quality in concrete, it is the logical choice for index to abrasion resistance.

CONCLUSIONS

1. Air content (or density) influences the resistance of concrete to abrasion but only in so far as it affects the compressive strength (Fig. 3) in other words air-entrained concrete is as resistant to abrasion as plain concrete providing they are of equal strength.

2. Compressive strength is the most important factor controlling the abrasion resistance of concrete (Fig. 3); abrasion resistance increasing as compressive strength increases (Fig. 4).

3. For concretes of equal air content the abrasion resistance is inversely proportional to water-cement ratio (Fig. 7).

4. Abrasion resistance and compressive strength vary inversely with voids-cement ratio, regardless of water-cement ratio, water, cement, or air contents (Figs. 8 and 9).

Acknowledgment:

The authors are indebted to Mr. W. H. Price, Head, Materials Laboratories, Research and Geology Division, for his helpful comments and suggestions.

The tests made in the Denver laboratories of the Bureau of Reclamation were under the direction of R. F. Blanks, Head, Research and Geology Division.

All design, construction, and research work of the Bureau are under the supervision of L. N. McClellan and all activities of the Bureau are under the direction of Michael W. Straus, Commissioner.

REFERENCES

(1) D. A. Abrams, "Wear Test of Concrete," *Proceedings*, Am. Soc. Testings Mats., Vol. 21, p. 1013 (1921).

(2) C. H. Scholer and H. Allen, "Wear Tests of Concrete," *Bulletin No. 20*, Engineering Experiment Station, Kansas State College, Manhattan, Kansas (1928).

(3) W. H. Price, "Erosion of Concrete by Cavitation and Solids in Flowing Water," *Journal* Am. Concrete Inst. *Proceedings*, May, 1947, Vol. 43, p. 1009.

(4) L. Schuman and J. Tucker, Jr., "A Portable Apparatus for Determining the Relative Wear Resistance of Concrete Floors," Research Paper No. RP1252, Nat. Bureau Standards, Washington, D. C. (1939).

(5) H. S. Meissner and S. E. Smith, "Concrete Curing Compounds," *Proceedings*, Am. Concrete Inst. p. 557, (1938).

(6) W. E. Emley and C. E. Hofer, "Test of Floor Coverings for Post Office Workrooms," *Journal of Research*, Nat. Bureau Standards, Vol. 19, November, 1937.

(7) H. L. Kennedy, "Homogeneity of Air-entraining Concrete," *Proceedings*, Am. Concrete Inst. Vol. 42, (1946).

(8) Ahlers Lindon, and Bird, "Wear Tests on Floor Finishes at Warehouse of R. H. Macy Company, Long Island City, New York," p. 778 (1929).

DISCUSSION

Mr. C. F. Mohr.[1]—Would not the aggregate have some effect on the results of wear of concrete if there was a 10 per cent or 15 per cent difference in Los Angeles wear test of the aggregates in the concrete?

Mr. R. C. Mielenz.[2]—I should think that the abrasion resistance of the aggregate would influence the abrasion resistance of the concrete, but I am convinced that the basic principles which we hope to advance here with regard to the influence of the void-cement ratio are of primary importance. With regard to the particular aggregate, it is the local aggregate from the Platte River which we customarily use in Denver for our laboratory control aggregate. Mr. Price might check me on this, but I believe its Los Angeles abrasion loss in 500 cycles will be usually around 30 per cent per weight.

Mr. W. H. Price.[3]—Forty.

Mr. Mielenz.—It was not a high-quality aggregate.

Mr. Bryant Mather.[4]—Since the question of aggregate has been raised, I wonder if Mr. Mielenz would agree that, in making abrasion tests of concrete, there are two quite different approaches, one of which involves the properties of the aggregate and the other does not. What I am driving at is this: if your abrasion study is concerned with the resistance of the surface and does not get down far enough into the concrete to become involved with the aggregate, you are dealing, as in this paper, largely with the properties of the matrix.

If you are concerned with abrasion of concrete to depth, then, obviously, the properties of the coarse aggregate are important. From the standpoint of designing an experiment on abrasion of concrete, it has seemed to us that that distinction is very important. There are, as was pointed out in the paper, many approaches to abrasion testing, and I think they may be classified at least roughly in terms of whether or not the abrasion is carried to such depth as to materially involve the aggregate.

Mr. W. H. Price.—A number of years ago I wrote a paper on abrasion of concrete and I searched the literature to see what I could find regarding tests which would indicate the effect of aggregates on this property of concrete. I did not find very much. I concluded from that search that aggregates do have an effect on the abrasion resistance of concrete but that it is secondary to the strength of the concrete.

Mr. Mielenz.—I would like to go on just a moment with this same line of thought. As you have noted from the slides illustrating abrasion to greater depths below the surface, aggregate particles commonly project above the general level of the pit. Consequently, if similar attrition were to occur on a floor or a surface subjected to abrasion, the abrasive action ultimately would be expended on projecting aggregate particles and, consequently, the characteristics of the cement paste would apparently be of considerably lesser significance.

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[2] Head, Petrographic Lab., U. S. Bureau of Reclamation, Denver, Colo.

[3] Materials Engineer, Bureau of Reclamation, Denver, Colo.

[4] Engineer (Concrete Research), Concrete Research Div., Waterways Experiment Station, Jackson, Miss.

However, I point out that the quality of the cement paste still would determine the tenacity with which those projecting aggregate particles were held in place, and if the paste were weak because of a high water-cement ratio, void-cement ratio or other conditions, then those aggregate particles would be more easily removed, even though they are hard themselves.

MR. R. E. DAVIS.[5]—I think we ought to realize that there are different types of abrasion. The cutting action of sand in rapidly running water, for example, is quite different from the rubbing or grinding action under the steel wheels of trucks on a floor; and while I am in entire agreement with the method that Mr. Mielenz has employed here as being quite suitable for the former condition, I do not believe it is a suitable test for the latter.

I am convinced, from fairly exhaustive investigations which we have carried out over the years, that the water-cement ratio of the paste plays a very important part in the wearing resistance of a floor, particularly initial wear on the always present surface film of hardened paste. When the wear becomes deeper, under the rolling action of truck wheels, then the quality of the exposed aggregate becomes the important factor.

MR. PRICE.—There are some very interesting data on wear published in *Bulletin No. 2* of the Lewis Institute, by Mr. Abrams in May, 1919. Concrete blocks 8 in. square and 5 in. thick were tested by Mr. Abrams by placing them as a liner for a drum 36 in. in diameter and subjecting them to the grinding action of 200 lb of cast iron balls for 1800 revolutions. The relation of strength to wear resistance obtained by Mr. Abrams is very similar to that reported in the present paper.

MR. NORMAN H. WITHEY, JR.[6]—I am wondering if the percentage of sand was progressively decreased with increase in air. I presume it was.

MR. MIELENZ.—It was held constant at 43 per cent.

MR. WITHEY.—Would that be done in practice? Your water-cement ratios might be considerably more reduced with an increase in air by reducing the percentage of sand as you went up in air; and if the water-cement ratio is a factor in resistance to abrasion it might affect the results rather appreciably.

MR. PRICE.—Entrained air improves the workability of concrete and for a given cement content the amount of sand used in the mix can be reduced in proportion to the percentage of air entrained. This reduction in sand reduces the amount of water required to produce a given slump with consequent lowering of the water-cement ratio and improvement in strength. Concrete of equal strength can be obtained by increasing the cement content as was done in the tests under discussion. It is agreed that it would have been more representative of what is done on the job to have reduced the percentage of sand as the percentage of air was increased, but I do not believe the conclusions would have been any different.

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[5] Professor of Civil Engineering, and Director, Engineering Materials Lab., University of Californa, Berkeley, Calif.

[6] Technical Representative, Combustion By-Products Co., Chicago, Ill.

PERMEABILITY TESTS OF LEAN MASS CONCRETE*

By Herbert K. Cook[1]

Synopsis

The use of air entrainment in mass concrete for large dams has made it possible to place concrete with cement factors as low as two bags per cu yd. Adequate strengths can be obtained with such concretes, but more information is desirable on the permeability of these concretes under relatively high hydrostatic pressures. The design and operation of permeability test equipment for testing 14½-diam by 15-in. cylindrical specimens at a pressure of 200 psi is described. Permeability coefficients of specimens representing nine air-entrained concretes containing 2½-in. aggregate but with water-cement ratios equal to those for full mass mixes with 6-in. aggregate and with cement factors of from 2.00 to 3.00 bags per cu yd are reported after testing at ages of 3 months, 1 yr, and 18 months. Plans for testing at later ages and for the testing of other specimens are described.

The Corps of Engineers has, with the use of air entrainment, successfully placed large volumes of concrete in the interior of several of its dams with a cement factor of 2½ bags per cu yd, and has experimentally placed concrete on the job with a cement factor of 2 bags per cu yd. The use of as low a cement factor as possible in this type of structure is highly desirable from the standpoint of economy and reduction in heat generation with consequent reduction in volume change. Adequate strength can be readily attained. However, to permit further economies in the design of such structures it is necessary, among other things, that additional information be made available on the permeability of these lean mass concretes, particularly at later ages, after filling of the reservoir.

Considerable literature is available on the general subject of the permeability of concrete, but only a relatively small portion of it deals with the permeability of mass concrete, and with the exception of the data given by Davis (**1**)[2] and Steele (**2**), which are largely reported herein, the author has been unable to obtain additional references to work on this subject on concretes with cement factors lower than four bags per cu yd.

The work by Ruettgers, Vidal, and Wing (**3**), which was done on concrete containing four bags of cement per cubic yard and with somewhat similar equipment, is apparently most closely comparable to that reported herein. Other work on the general subject and comments thereon are contained in References (**4**) through (**11**).

The design and construction of the permeability test equipment, the fabrication of the test specimens, and permeability measurements at an age of three months were performed under the supervision of Raymond E. Davis at

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* Presented at the Fifty-fourth Annual Meeting of the Society, June 18–22, 1951.

[1] Chief, Concrete Research Division, Waterways Experiment Station, Corps of Engineers, U. S. Army, Jackson, Miss.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1164.

TABLE I.—GRADING OF SANDS.

Sieve	Individual Per Cent Retained		
	Coarse	Medium	Fine
No. 4	0	0	0
No. 8	15.0	11.0	8.4
No. 16	17.0	14.0	11.5
No. 30	26.0	23.0	22.9
No. 50	25.0	26.0	25.5
No. 100	14.0	18.0	17.7
No. 200	2.25	6.0	10.5
Pan	0.75	2.0	3.5
Fineness Modulus	2.85	2.50	2.25

TABLE II.—GRADING OF GRAVEL.

Sieve	Per Cent Retained	
	Individual	Cumulative
1½ in.	39.6	39.6
¾ in.	36.2	75.8
½ in.	12.2	88.0
⅜ in.	6.0	94.0
No. 4	6.0	100.0
Fineness Modulus	...	8.09

TABLE III.—CONCRETE MIXTURES AND PERMEABILITY TEST RESULTS.

Mixture Number	*A*	*B*	*C*	*D*	*E*	*F*	*G*	*H*	*I*	*J*	*K*	*L*[g]	*M*[h]
Cement factor (mass) bags per cu yd[a]	2.00	2.00	2.25	2.25	2.25	2.25	2.50	2.50	3.00	2.25	2.25	2.25	...
Cement factor (lab) bags per cu yd[b]	2.48	2.49	2.78	2.76	2.73	2.72	3.01	3.00	3.74				
Sand, fineness modulus	2.50	2.25	2.25	2.85	2.50	2.25	2.85	2.50	2.85	2.25	2.25	2.25	0.15
Sand, per cent of agg. (mass)[a]	18.00	18.00	17.5	17.5	17.5	17.5	17.0	17.0	16.0				...
Sand, per cent of agg. (lab)[b]	25.75	25.75	25.00	25.00	25.00	25.00	24.25	24.25	22.75				...
Water-cement ratio, by weight	0.80	0.86	0.84	0.67	0.72	0.76	0.60	0.66	0.50	0.83	0.69	0.76	0.80
Air content of concrete finer than 1½ in., per cent	6.4	6.2	1.4	6.2	6.2	6.2	6.4	6.4	6.2	6.2	5.7	6.5	3.9
Slump, in.	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5	3.3	0.2	1.3	...
PERMEABILITY COEFFICIENT $K_c \times 10^{12}$[c]													
3-month age	1210	810	715	72[f]	364	397	866	276	1123[f]	219	481	3740	3970
	1150	413	1820[f]	1760[f]	1610	705	665	901	240	301	84	...	2290
	231	[e]	2310[f]	843	703	145	563	803	117	731	364	...	...
	444	411	943	576	539	588	423	754	161	369	203	...	...
	388	283	329	1359	936	360	353	703	280	567	717	...	...
Average	685	479	662	926	830	439	574	687	200	437	370	3740	3130
1 yr age[d]	124	205	84	51	173	154	142	38	77	130	33	116	390
	185	105	467	208	229	107	142	64	63	108	23	...	219
	74	[e]	252	96	119	91	112	117	47	78	70	...	...
	152	229	145	124	257	210	168	115	28	...	26	...	...
	129	33	121	121	360	219	131	83	65	153	...	...	...
Average	133	143	214	120	228	156	139	83	56	117	38	116	304
1½-yr age[d]	459	479	275	64	224	70	422	116	231	...	...	...	...
	228	458	524	157	257	82	76	210	278	...	...	...	...
	815	656	368	121	266	72	255	115	140	...	...	...	...
	109	186	409	111	149	89	98	183	248	...	...	...	...
	129	313	481	77	184	149	159	76	119	...	...	...	...
Average	348	418	411	106	216	92	202	140	203	...	...	...	...
3-month age (Vacuum-saturated specimens)	...	793	...	...	...	...	...	...	154	...	...	...	...
	...	612	...	...	...	...	...	...	423	...	...	...	...
	...	705	...	...	...	...	...	...	514	...	...	...	...
	...	1331	...	...	...	...	...	...	789	...	...	...	...
	...	787	...	...	...	...	...	...	1030	...	...	...	...
Average	...	846	...	...	...	...	...	...	582	...	...	...	...
1-yr age[d] (vacuum-saturated specimens)	...	341	...	...	...	...	...	...	37	...	...	...	...
	...	357	...	...	...	...	...	...	37	...	...	...	...
	...	315	...	...	...	...	...	...	47	...	...	...	...
	...	262	...	...	...	...	...	...	156	...	...	...	...
	...	257	...	...	...	...	...	...	82	...	...	...	...
Average	...	306	...	...	...	...	...	...	72	...	...	...	...

[a] Data for mass mixture with 6-in. aggregate obtained by adding the solid volume of the 3- to 6-in. aggregate to the laboratory mixture containing 2½-in. aggregate, and allowing for a small increase in the volume of cement paste required to maintain a slump of 1½ in. for mixtures *A* through *I*.

[b] Laboratory mixtures containing 2½-in. aggregate, used to make test specimens except for mix *M*.

[c] Permeability coefficient $K_c = cfs/ft^2$ (*ft head per ft*) at a test pressure of 200 psi. Tests at 3-month age by Raymond E. Davis, tests at later ages made at Waterways Experiment Station.

[d] Tests at 1-yr age made on specimens that were tested at 3-month age, tests at 1½-yr age made on specimens not previously tested, additional specimens representing mixtures *A* through *I* will be tested at 2-yr and 5-yr ages.

[e] Specimen broken.

[f] Omitted from average.

[g] Crushed quartzite aggregate.

[h] Mortar grout.

Berkeley, Calif., for the Office, Chief of Engineers.

The basic permeability rigs and all of the test specimens were transferred after completion of the 3-month tests to the Concrete Research Division of the Waterways Experiment Station at Jackson, Miss., where tests at the 1-yr age were made and at which tests will be made at later ages.

This paper discusses the major items of the work in the following order: (1) the concrete materials and the design of the concrete mixtures, (2) the fabrication and storage of the specimens, including a summary of the specimens made, (3) a description of the permeability equipment and the test procedure, (4) a discussion of the results obtained, (5) comments on plans for future work.

CONCRETE MATERIALS AND DESIGN OF CONCRETE MIXTURES

Concrete Materials:

The cement complied with the requirements of ASTM Specification C 150 for Type II portland cement[3] and had a low-alkali content. The air-entraining agent in all mixes except mix C was neutralized Vinsol resin. The sand and gravel were obtained from sources which are being used in the construction of the Pine Flat Dam, on the Kings River, California.

The fine aggregate was processed into seven sizes. The size fractions were batched individually to produce the gradings shown in Table I, which were used in the various laboratory mixes as indicated in Table III.

The coarse aggregate was of 2½-in. maximum size and was processed into five sizes which were batched individually for all laboratory mixes to produce the grading of coarse aggregate shown in Table II.

Concrete Mixtures:

The principal characteristics of the mixtures used in the tests are shown in the top part of Table III. The basic mixtures are designated A to I. A number of tests were made on special mixtures designated J to M.

The following discussion is applicable only to the basic mixtures *A* to *I*. Where reference is made to "mass mixtures," concrete containing 6-in. aggregate is implied, and reference to "laboratory mixtures" implies concrete containing aggregate no larger than 2½-in. The base mass mixture A was made at the Waterways Experiment Station. A program of trial mixtures was conducted at Berkeley to determine, for the nine basic laboratory mixtures, the proper proportions to use in casting laboratory specimens which would contain 2½-in. aggregate instead of 6-in. but which would have the same water-cement ratio, air content, and slump of wet-screened concrete as the nine mass mixtures.

First, it was necessary to establish the water-content ratio which would produce the desired slump for the eight mass mixtures B to I. It was found by trial that for each 0.25 bags per cu yd over the cement content of mixture A, an increase of 2 lb of water per cu yd was required to maintain the fixed consistency. The water contents of mixtures E and H, which were like mixture A except for cement content, were thus established and are shown in Table III. The water content of a theoretical mixture "X" was established in the same manner. This mixture was not actually made, but the proportions were required to establish mixture I.

Second, the cement-aggregate ratios for laboratory mixtures A, E, H, and a laboratory mixture based on theoretical mixture "X" were established by trial, with the respective water-cement ratios maintained equal to those of the corresponding mass mixtures but with the paste content varied to produce the fixed consistency.

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[3] Specification for Portland Cement (C 150 – 49), 1949 Book of ASTM Standards, Part 3, p. 1.

Third, the water-cement ratios for the remaining six laboratory mixtures (B, C, D, F, G, I) were established by trial, each with the same aggregate-cement ratio as that of the mixtures (A, E, H, or X) having the same cement content, but with the water content varied to produce the fixed consistency. The resulting water-cement ratios are given in Table III. Table III also shows the actual cement content for each laboratory mixture. The values are averages of the several batches actually made for manufacture of the specimens.

Fabrication of Specimens:

The permeability specimens were 14½-in. diameter by 15-in. cylinders. The materials were pre-cooled to produce a concrete temperature of approximately 50 F at the time of mixing. The concrete was mixed in batches of 500 to 550 lb for 3 min in a 3½-S commercial tilting mixer. The specimens were molded in a room maintained at 50 F.

The forms were of No. 26-gage galvanized sheet steel with integral bottoms. All seams were crimped and soldered. A 0.25-in. flange was turned out around the top, and a cover was provided having a diameter of 0.25 in. less than the outside diameter of the flange. The flange and the contacting surface of the cover were tinned to facilitate soldering the cover in place after molding the specimen. Semi-cylindrical form stiffeners of 0.25-in. steel plate were bolted together around the thin sheet-metal form while a specimen was being cast. Pairs of 1 by 16 by 16-in. plywood plates and four ⅜-in. tie rods were used with each form stiffener to tie the tops and bottoms of the forms together when turning the specimens on their sides.

Some of the mortar was placed around the bottom corner of the form and the form was then filled with concrete in two layers, vibrating each layer. The top of the specimen was screeded and a small depression made around the top edge to prevent water from interfering with the soldering operation. The top cover was immediately soldered on and checked for leaks.

Immediately after sealing, the top plywood plate was bolted to the bottom plate and the specimen turned on its side so that water gain would be normal to the axis of the specimen and bleeding channels would be oriented as they would in the upstream face of a dam. Since the specimens were to be tested by applying water pressure to the circular end, it was necessary to turn the specimen on its side while bleeding could occur.

Storage of Specimens:

All specimens were stored at 50 F for 2 days, when the form stiffeners were removed. They were then moved to storage at 70 F until the age of 7 days, and then to storage at 80 F. This procedure was followed to simulate mass curing.

The specimens for test at the age of 3 months were removed from 80 F storage at the end of 2 months, were stripped of their metal containers, and were stored at 70 F. The top and bottom of the specimens were sandblasted to remove their surface skin and were coated on their cylindrical surfaces with a 70-30 paraffin-rosin mixture, and were then stored in water to a depth of four in. to aid in their saturation. The top surface as cast, which later was to be the top during the permeability test, was placed on the bottom so that the air within the specimen might be forced out the top (the bottom as cast) as partial saturation by capillarity occurred.

The specimens for test at ages later than 3 months were removed from 80 F storage at 3 months and were stored at 70 F in their sealed containers.

After completion of the permeability tests at the age of 3 months, the specimens so tested were placed in moist storage at 70 F for retesting at 1-year age.

All of the permeability specimens and the high-pressure permeability rigs with calibrated reservoirs were shipped on May 5, 1950, in a carload lot to the Concrete Research Division. The specimens which had been tested at 3 months were packed in wet sawdust and sealed in building paper in one end of the car. Since the specimens for test at later ages were still in their sealed containers, this precaution was not necessary for them. Shipment was made *via* the southern route and normal spring temperatures prevailed throughout the shipment. The specimens in wet sawdust were still damp and were placed in moist storage at the new standard temperature of 73.4 ± 2 F, as were the sealed specimens.

Summary of Specimens:

A total of 205 permeability specimens were manufactured of which 2 were exploratory, 3 were broken, and 200 are to be tested by the Waterways Experiment Station. A summary of the number of specimens that have been or are to be tested at the various ages is given below:

Number tested at Berkeley at 3-month age	67	
Number retested at WES at 1-yr age	65	
WES sub-total		65
Number to be tested at WES at $1\frac{1}{2}$-yr age		45
Number to be tested at WES at 2-yr age.		45
Number to be tested at WES at 5-yr age.		45
Total for test at WES		200

Each of the basic mixtures, A to I, is represented by 20 specimens of which 5 are to be tested at each age, except for the 3-month and 1-yr tests which were made on the same specimens. Mixture B was represented for the 3-month and 1-yr tests by only 4 specimens. In addition five extra specimens were made from both mixes B and I. These specimens were tested at 3 months and retested at 1 yr only to determine whether the relatively rapid saturation by vacuum and pressure would affect the permeabilities as compared to the relatively slow and perhaps incomplete saturation by capillarity and pressure employed for the principal tests.

Special mixtures J and K, representing wet and dry consistencies, were represented by five specimens each for test at 3 months and retest at 1 year only; however one specimen from each mixture was broken between 3 months and 1 yr.

Special mixture L contained crushed quartzite for both coarse and fine aggregate and was represented by only one specimen which was tested at 3 months and retested at 1 yr.

Special mixture M was 1:1.75 cement-sand grout containing a very fine sand of which 60 per cent passed the No. 200 sieve. This condition was represented by two specimens which were tested at 3 months and retested at 1 yr.

DESCRIPTION OF PERMEABILITY EQUIPMENT AND TEST PROCEDURE

Permeability Equipment:

A schematic drawing of the test assembly is shown in Fig. 1. The specimens were mounted in the container as shown in the figure. The inside diameter of the container was $\frac{7}{8}$ in. greater than the outside diameter of the specimen. Both the cylindrical surface of the specimen and the inside cylindrical surface of the container were painted with a hot paraffin-rosin compound before the specimen was placed in the container. The specimen was bedded on a retainer ring, at the lower end of the container, with hydrostone, a gypsum product of high porosity as compared with that of concrete. After the specimen was bedded, a 1-in. layer of paraffin-rosin seal was placed just above the hydrostone. The remaining annular space between the specimen and the side of the container was filled with hot 200–300 penetration asphalt placed by means of a pressure pot. After the asphalt had cooled and shrunk, the depression thus formed was filled with the paraffin-rosin compound to prevent the oil in the asphalt from mixing with the water during test. The top and bottom of the container were then bolted securely in place, pressure-tightness at the top being made with a gasket, and all pipe connections made up.

The desired water pressures were produced by compressed air applied to the water surface in a calibrated reservoir of standard 2-in. pipe, and the inflow was periodically measured by the level of the water surface as indicated by a gage glass connected to the reservoir.

Representative of conditions likely to be found in the headwater of a dam, the water supply was tap water, and was air-saturated at atmospheric pressure. It was considered essential, in determining permeability, that the air content of water reaching the specimen under pressure should not exceed that of saturation at atmospheric pressure; otherwise as the pressure decreased from the test pressure of 200 psi at the pressure face to 0 psi at the bottom face it was thought that air

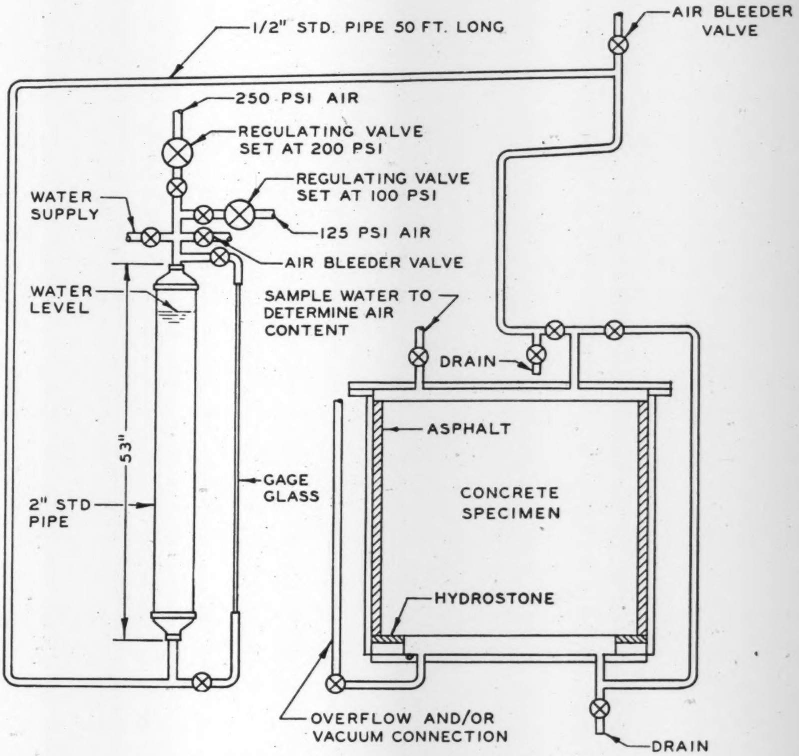
1/2" STD. PIPE 50 FT. LONG
AIR BLEEDER VALVE
250 PSI AIR
REGULATING VALVE SET AT 200 PSI
REGULATING VALVE SET AT 100 PSI
WATER SUPPLY
125 PSI AIR
AIR BLEEDER VALVE
WATER LEVEL
SAMPLE WATER TO DETERMINE AIR CONTENT
DRAIN
ASPHALT
53"
GAGE GLASS
CONCRETE SPECIMEN
2" STD PIPE
HYDROSTONE
OVERFLOW AND/OR VACUUM CONNECTION
DRAIN

FIG. 1.—Permeability Test Assembly (Schematic).

would be liberated within the pores and capillaries which would act to decrease the rate of flow.

As the application of air pressure to the water would cause it to absorb air which would gradually diffuse through the system, a long supply line (approximately 50 ft) was installed between the calibrated reservoir and the container in which the specimen was mounted. Exploratory tests showed that even under high pressures the air diffuses so slowly through water that the long supply line prevented water with excess air content from reaching the specimen. Frequent determinations of the air content of the water at the specimen were also made, and the water was drained and replenished whenever the air content exceeded 0.2 per cent. The photograph in Fig. 2 shows a part of the actual installation of 15 rigs at the Waterways Experiment Station. The copper coil immediately above the specimen container makes up the 50 ft of supply line for the purpose of preventing excess air in the water from reaching the specimen.

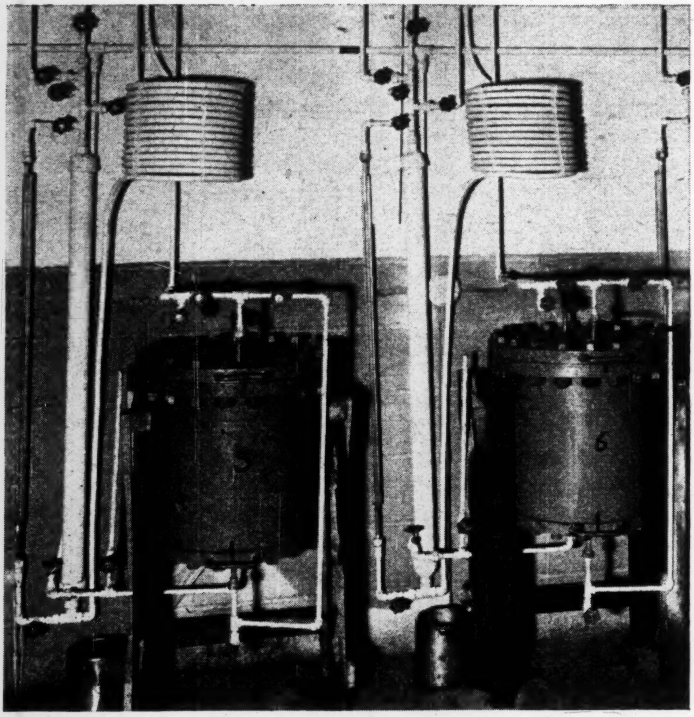

FIG. 2.—Permeability Apparatus.

Test Procedure:

In making a permeability test, the system is first cleared of all water by opening all outlet valves and blowing air through the system. A vacuum is then applied to the vacuum connection, and the entire system is exhausted to a vacuum of 28 in. of mercury. The water valve is opened and the system filled with water. The air bleeder valve opposite the water valve is opened until water flows and air is also flushed out at the high-point air bleeder valve.

When the entire system is free of air and bleeder valves closed, 100-psi air pressure is applied to the reservoir and held for 5 min. The air pressure is then increased to 200 psi. The water level as indicated by the gage reading is determined each day until the flow becomes constant. This normally requires about two weeks.

The permeability coefficient, K_c, is calculated from the following formula:

$$K_c = \frac{cfs}{\text{ft}^2(\text{ft head per ft})}$$

Considerable time was spent eliminating leaks in the pipe connections and valves when the equipment was first placed in operation at each location, but once these difficulties were overcome, very little trouble was experienced in the operation of the equipment, and the detection and correction of the leaks in the system is a minor problem.

DISCUSSION OF TEST RESULTS

A large part of the work is yet to be completed; therefore this discussion will be confined to the results obtained on the specimens tested at 3 months and retested at 1 year, and to the specimens which remained sealed until the 1½-yr tests. Any conclusions are entirely tentative and should be applied only to the specimens tested. The results do however help to verify previous work by others, and while there is the usual relatively high variation between individual specimens representing one test condition the average results for the series appear to indicate quite definite trends. One fact that should be kept in mind is that while every attempt was made to have the laboratory mixtures represent lean mass concrete they did contain only 2½-in. aggregate and not 6-in. aggregate. It is felt that the mortar phase of the concretes are truly representative of mass concrete even though the 6-in. aggregate was absent and the bond conditions which exist under such cobbles were not represented. It is believed that representative permeability results cannot be obtained on such concrete unless a minimum specimen diameter of 30 in. is used. It was not possible to investigate actual mass concrete since neither funds nor equipment for fabricating and testing such large specimens were available.

The results of all completed permeability tests are shown in the lower part of Table III and include results obtained at ages of 3 months, 1 yr, and 1½ yr.

Basic Mixtures:

The permeability of the specimens retested at 1 yr was very considerably lower than the permeability of the same specimens when originally tested at 3 months. This was to be expected, since hydration of the cement, assisted by saturation of the specimens, had continued for an additional 9 months.

The average results obtained on specimens tested at 1½ yr were considerably lower than the tests on corresponding specimens at 3 months except for mix *I*. The results were higher in some cases at 1½ yr than for the 1-yr retests on the specimens originally tested at 3 months. The fact that the 1½-yr specimens were not previously saturated under pressure undoubtedly delayed their hydration rate as compared to the saturated specimens retested at 1 yr. Some of the groups, however, apparently overcame this delay in the extra 6 months of aging since some did show less permeability at 1½ yr than at 1 yr. The differential between 1 and 1½ yr was in general considerably less than between 3 months and 1 yr.

Considering individual specimens in the respective groups representing the basic mixtures, the specimen that was high or low in the group when tested at 3 months was, with but few exceptions, also high or low in the group when retested at 1 yr. The fact that the individual specimens maintained their general order of position in the group indicates that leakage or other inaccuracy in the testing was not a source of major error. The deviation of individual specimens from the average for the group was much lower in the 1-yr tests and generally lower for the 1½-yr tests than for the 3-month tests. A partial explanation for this is that the rate of hydration of the cement had decreased and the several specimens for the group had levelled off at conditions more nearly approaching equilibrium than was the case at 3 months.

Special Mixtures or Conditions:

It appears from a comparison of the vacuum-saturated specimens with the pressure-saturated specimens for mixtures B and I that vacuum-saturation increases the permeability coefficient.

The relatively wet concrete having a slump of 3.3 in. (mixture J) was not greatly different in permeability as compared with concrete having a slump of 1.5 in., but the relatively dry concrete having a slump of 0.2 in. (mixture K) had a lower permeability.

The permeability coefficients for both mixture L (quartzite aggregate) and mixture M (cement grout) were far greater at 3-months age than any of the other concretes tested but showed a greater reduction when retested at 1 yr than any of the other concretes.

PLANS FOR FUTURE WORK

The tests already scheduled for 2- and 5-yr ages will be carried out on the specimens now in storage for such tests. It is possible that some of the specimens already tested or scheduled for test will be retested at later ages.

A total of 960 6 by 6-in. specimens containing ¾-in. aggregate and representing a large number of concrete mixtures, particularly blends of portland cement with other hydraulic, pozzolanic, or other finely divided materials are scheduled for fabrication within the next year. These specimens will be tested for permeability at 3 months, 1 yr and *N* yr. It is proposed to make additional 14 ½-diameter by 15-in. specimens on a number of the more important of the above concretes. These specimens will contain 2½-in. aggregate. It is also proposed, at a later date, to make 30-in. diameter specimens on a relatively few of the most important of the above concretes. These 30-in. specimens will contain 6-in. aggregate and will be full-scale mass concrete mixtures. It is probable that the full-scale mass concrete specimens will be drilled from 10- by 10-ft blocks made with concrete produced in a commercial 2-yd batching and mixing plant.

REFERENCES

(1) R. E. Davis, "Permeability and Triaxial Tests of Lean Mass Concrete," a report to the Office, Chief of Engineers, Corps of Engineers, U. S. Army, Washington, D. C., April 1950.

(2) B. W. Steele, "Concrete in Large Dams," paper presented at the Fourth World Power Conference at New Delhi, India, January, 1951.

(3) A. Ruettgers, E. N. Vidal, and S. P. Wing, "An Investigation of the Permeability of Mass Concrete with Particular Reference to Boulder Dam," *Journal*, Am. Concrete Inst. *Proceedings*, Vol. 31, p. 382 (1935).

(4) J. W. Kelly, "Some Time-Temperature Effects in Mass Concrete," *Journal*, Am. Concrete Inst., *Proceedings*, Vol. 34, p. 573 (1938).

(5) H. F. Gonnerman, Chairman, "Current Researches on Plain and Reinforced Con-

crete and Related Materials—Report of Committee 101, Survey of Concrete Research," *Journal*, Am. Concrete Inst. *Proceedings*, Vol. 27, p. 469 (1931).

(6) G. W. Washa, "The Efficiency of Surface Treatments on the Permeability of Concrete," *Journal*, Am. Concrete Inst. *Proceedings*, Vol. 30, p. 1 (1934).

(7) F. R. McMillan and I. Lyse, "Some Permeability Studies of Concrete," *Journal*, Am. Concrete Inst. *Proceedings*, Vol. 26, p. 101 (1930).

(8) P. T. Norton and D. H. Pletta, "The Permeability of Gravel Concrete," *Journal*, Am. Concrete Inst. *Proceedings*, Vol. 27, p. 1093 (1931).

(9) G. W. Washa, "Comparison of the Physical and Mechanical Properties of Hand Rodded and Vibrated Concrete Made with Different Cements," *Journal*, Am. Concrete Inst. *Proceedings*, Vol. 36, p. 617 (1940).

(10) W. M. Dunagan, "Methods for Measuring the Passage of Water Through Concrete," *Proceedings*, Am. Soc. Testing Mats., Vol. 39, p. 866 (1939).

(11) R. E. Davis, "A Review of Pozzolanic Materials and Their Use in Concretes," Symposium on Use of Pozzolanic Materials in Mortars and Concretes, p. 3, Am. Soc. Testing Mats. (1949). (Symposium issued as separate publication, *STP No. 99*.)

USE OF THE SONISCOPE FOR MEASURING SETTING TIME OF CONCRETE*

BY E. A. WHITEHURST[1]

SYNOPSIS

The soniscope has been used to test concrete specimens and concrete in place for the past several years. During the past year, efforts have been made to study the changes occurring in fresh concrete during its setting period by measuring the velocity of propagation of small mechanical impulses through the concrete. Very stiff concrete, $\frac{1}{8}$ to $\frac{1}{4}$-in. slump, was cast into 4 by 4 by 16-in. forms. Immediately after placing, the endplates of the forms were removed. With the stiff mixes used no slumping of the specimen ends was observed. Velocity tests were made periodically, using the soniscope, from shortly after the specimens were cast until 8 hr or longer had elapsed.

It was observed that the initial velocities were quite low, in the order of 4000 ft per sec. During the early hours, however, this velocity increased at a rapid rate. After a period of time varying from about $4\frac{1}{2}$ to $8\frac{1}{2}$ hr, the rate of increase in velocity suddenly changed, the increase continuing at a much slower pace. The point, in time, at which this change occurred was taken as the time of set of the concrete.

Four cements, conforming to ASTM types I, II, III, and IV, were used in this study. Their times of set, measured by the Gillmore needles, were 5:45, 6:50, 4:15, and 8:00, respectively. The corresponding times of set of the concretes were 5:45, 7:10, 5:00 and 7:40. When the velocities indicated that the concrete had set, the specimens were sufficiently hard to be removed from the forms. A few tests have been made on concretes of more normal consistency (water-cement ratio = 0.5 by weight).

Although this method of test has not yet been applied to concrete in place and the work to date may be considered as largely exploratory, it is felt that it offers excellent possibilities for studying the behavior of green concrete and may, with experience, have considerable use in evaluating construction practices.

The need for a method of determining the setting time of concrete has long been apparent. Various methods have been proposed for the measurement of this property of a neat-cement paste. The ASTM has standardized and accepted two such methods, the Gillmore and Vicat needle methods, which are described in Standard Method of Test for Time of Setting of Hydraulic Cement by the Vicat and Gillmore Needles (C 191 - 49).[2] Recommendations for the use of variations of these tests involving mechanical application of the needles to reduce operator error have been proposed by Chisholm (**3**)[3] and Miller (**9**) and more

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[2] 1949 Book of ASTM Standards, Part 3, p. 149.

[3] The boldface numbers in parentheses refer to the list of references appended to this paper, see p.1176.

recently by Glantz and Halsted (5). Cement-sand mortars have also been tested in these automatic time-of-set devices.

Concrete, because of the large range in particle size, is not readily adaptable to a needle penetration test. In 1921, Davis (4) reported using a flow test for measuring setting time of concrete and mortar. A table similar to that used in determining normal consistency was employed. The time at which the concrete or mortar pat refused to flow was found to correspond to initial set. Davis suggested that early compression tests be used instead of a test for final set.

During the past few years some mention has been made of the possibility of measuring changes in the condition of green concrete through the use of a dynamic method of test—specifically, the measurement of the velocity with which an energy pulse travels through the concrete. Jones (6), in 1949, reported making such tests on laboratory specimens, using equipment built in the Road Research Laboratory, England. He stated, however, that below ages of 10 hr considerable difficulty was experienced in obtaining an adequate signal through the concrete. Arndt (1), reporting on tests made on concrete pavements in Kansas during the summer of 1949, showed data from tests made as early as 3½ hr after the concrete was placed. He did not comment upon the ease or difficulty with which the tests were made. The study currently reported was largely suggested by the foregoing.

The instrument used for measuring pulse velocity through concrete, in this study, was the soniscope. Leslie (7), Cheesman (2), and Leslie and Cheesman (8) have reported on the development and construction of this device and its use in Canada. The author (10) has shown results of its use in testing concrete structures in the United States. Although no complete description of the apparatus is offered at this time, it may be briefly described as a piezoelectric transmitter, a similar receiver, and the necessary electronic circuits to actuate the transmitter, amplify the received signal, and measure the transit time of a pulse going from the transmitter to the receiver. With this transit time and the distance between transducers known, the pulse velocity may be computed directly.

The purpose of this study was to investigate the possibility of determining the setting time of concrete by measurement of pulse velocities through the still plastic material. No effort was made to investigate the several variables which influence the time of setting of concrete. In so far as possible, these variables were minimized except where necessary to provide sufficient range in setting times to permit a satisfactory evaluation of the proposed test method.

DESIGN OF MIXES

In order to introduce into the study some element which would cause the time of set of the concretes to vary, preferably in some predetermined manner, and still keep all mixes as uniform as possible, it was decided that the mix design and method of handling should be kept constant, and one of the constituents of the mix should be varied. Since various portland cements conforming to ASTM types I, II, III, and IV could be procured, the type of cement used was, in most cases, the only variable between batches. Batches 1 through 8 were made from a 6-bag mix with a nominal water-cement ratio of 0.40 by weight. The reason for the very stiff mix is discussed under casting of the specimens. Actual water-cement ratios varied from 0.416 to 0.430 with slumps ranging from barely perceptible to $\frac{3}{8}$ in. Each of the four cements was used in two consecutive batches which were mixed on different days.

Toward the end of the study a few batches of a somewhat wetter consistency were made. Batches 9 and 10 were mixed with nominal water-cement ratios of 0.50 by weight. Types I and III cements were used in these batches, respectively, and slumps of 6½ and 2½ in. were measured.

The same aggregates, locally available glacial sand and gravel with satisfactory field performance records, were used throughout. The sand, as used, contained a small amount of free water, usually less than 1.5 per cent. The coarse aggregate was divided into four sizes and was stored in a rather dry condition. Before mixing, it was recombined and immersed in water overnight.

The concrete was mixed in a Lancaster tub mixer. Fine and coarse aggregates, together with part of the mixing water, were placed in the tub and mixed for 1 min. The remaining water and the cement were added, and mixing was resumed for an additional 5 min. The mixer blades were then raised from the tub and the slump and unit weight of the mix was determined.

CASTING OF SPECIMENS

Specimens were cast into 4 by 4 by 16-in. beams. Nine beams were cast from each 1.5-cu ft batch; three triple-section molds were employed. Specimens were molded, generally, in accordance with the ASTM requirements for the molding of laboratory specimens.[4] Because of the unusual stiffness of the plastic concrete, extensive rodding was required. The top surfaces of the beams were finished with a dampened wooden float, an operation which was rather difficult. Despite considerable effort to achieve well-compacted specimens, some honeycombing was noted in most cases.

The forms used were so constructed that single plates at each end served as

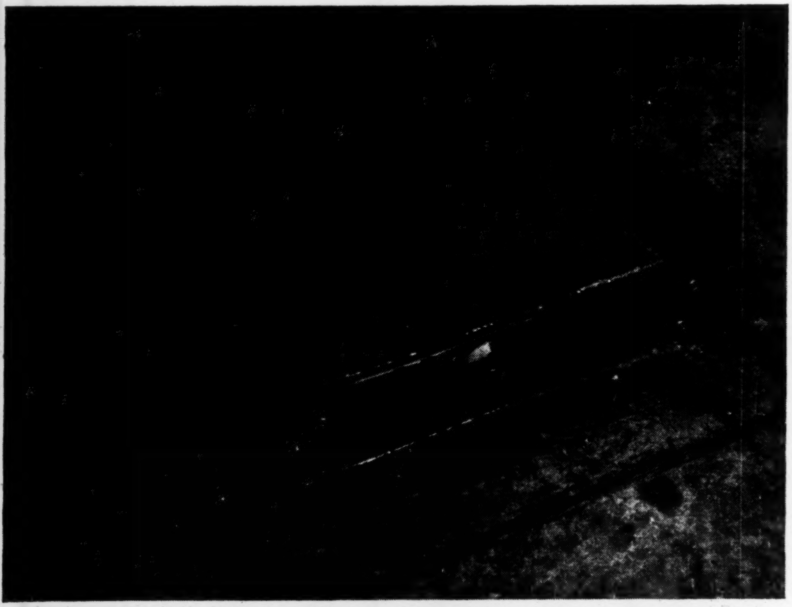

FIG. 1.—Specimens in Forms Immediately After Casting.

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[4] Standard Method of Making and Curing Concrete Compression and Flexure Test Specimens in the Laboratory (C 192 – 49), 1949 Book of ASTM Standards, Part 3, p. 836.

the end plates for all three beams. Shortly after the floating of the specimen surfaces was completed, these end plates were removed. The use of stiff mixes, permitting this practice, allowed early access to the ends of the beams for testing purposes. Figure 1 shows a mold in which three beams had just been cast; Fig. 2 was taken less than 5 min later, after the end plates had been removed. In the stiff batches, 1 through 8, little tendency of the ends of the beams to slump was noted. In the case of batches 9 and 10, the end plates were not removed for several hours, and no slumping of the beam ends was observed.

FIG. 2.—Specimens Shown With End Plate of Forms Removed.

TESTING OF SPECIMENS

Pulse velocity tests were begun on the beams as soon as possible after the removal of the end plates. Fig. 3 shows such a test in progress. The transducers were pressed against the exposed ends of a specimen, with a film of castor oil between the concrete and the rubber diaphragm of each transducer. The transit time of the vibration passing through the concrete, between transducers, was then measured. It was found that castor oil must be liberally applied to the transducer diaphragm, and in some cases directly to the ends of the beams, before a vibration could be successfully transmitted through the fresh concrete.

Initial velocity tests were made on the specimens in the first mold from 2 to 4 hr after the concrete was mixed, depending upon the type of cement used. Earlier tests were attempted but were found to be unsatisfactory. Once a satisfactory test was accomplished, the beams were tested repeatedly, usually at intervals of ½ hr.

It had been suggested that the vibrations passed through the specimen in testing, though of a minute nature, might

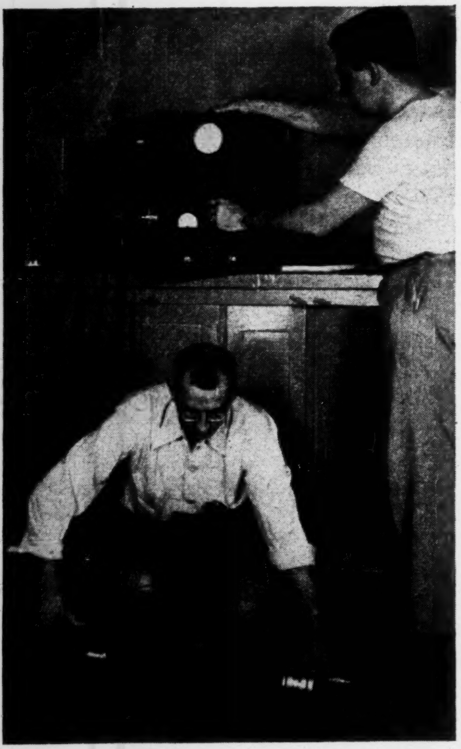

FIG. 3.—Testing Specimen With Soniscope.

have some material effect upon the concrete. To check this hypothesis, only the specimens cast in the first mold were tested throughout the entire setting period. Those in the second mold were subjected to their first velocity tests approximately 3 hr after those in the first mold, after the setting process was well under way. Tests were not begun on the beams in the third mold until it was felt that final set had occurred. Only a few tests were made on these specimens.

The half-hour velocity tests were continued until the specimens were 8 to 9 hr old and appeared to have reached final set. The beams were then allowed to remain in the forms over night. On the following morning, when the concrete was approximately 24 hr old, velocity tests were made on all nine of the specimens, the forms were stripped and the beams were moved to the moist room. Additional velocity tests were made when the specimens reached ages of 7, 14 and 28 days.

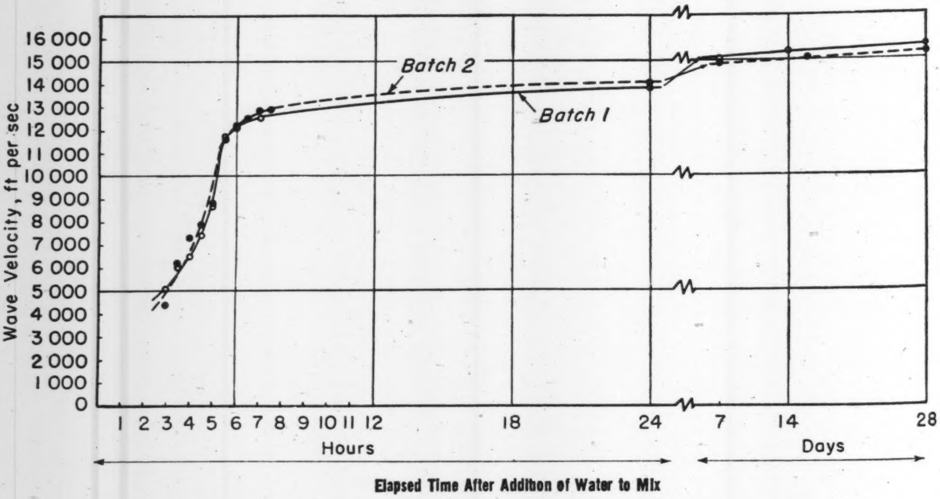

FIG. 4.—Average Wave Velocities Through Concrete Made From Type I Cement.

RESULTS AND DISCUSSION

In all cases it was found that the pulse velocity through the specimens increased at a rapid rate during the first few hours after the concrete was mixed. Figures 4, 5, 6, and 7 show the results of tests on mixes containing types I, II, III and IV cements, respectively. Each point represents the average of test values for the three beams cast in the first mold. It may be noted that velocity when first measured was in the range of 3000 to 6000 ft

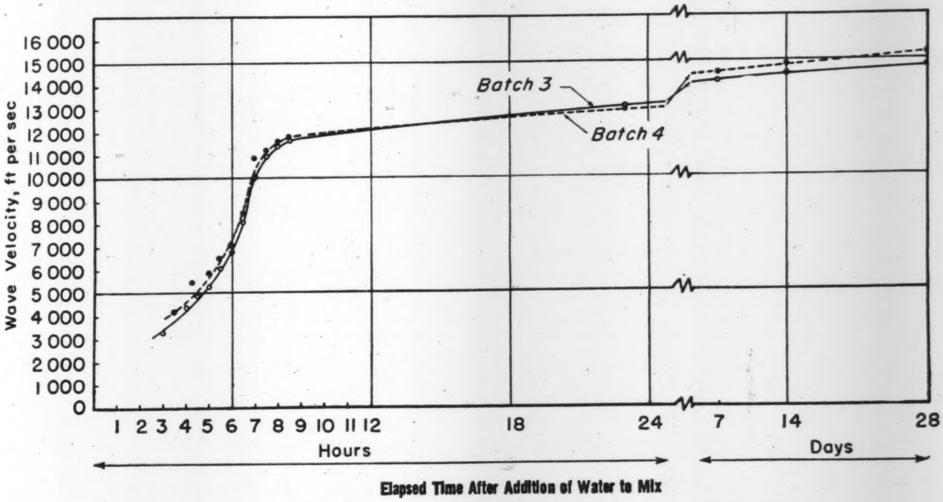

FIG. 5.—Average Wave Velocities Through Concrete Made From Type II Cement.

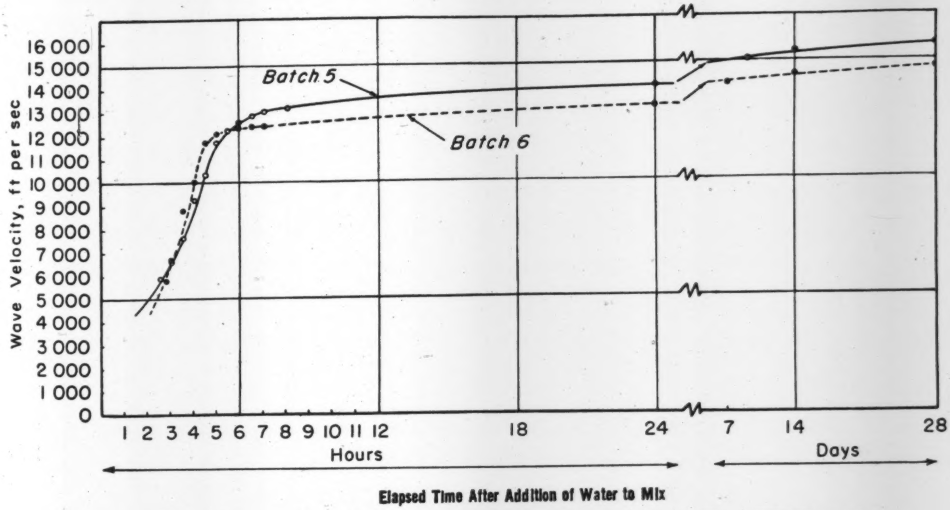

FIG. 6.—Average Wave Velocities Through Concrete Made From Type III Cement.

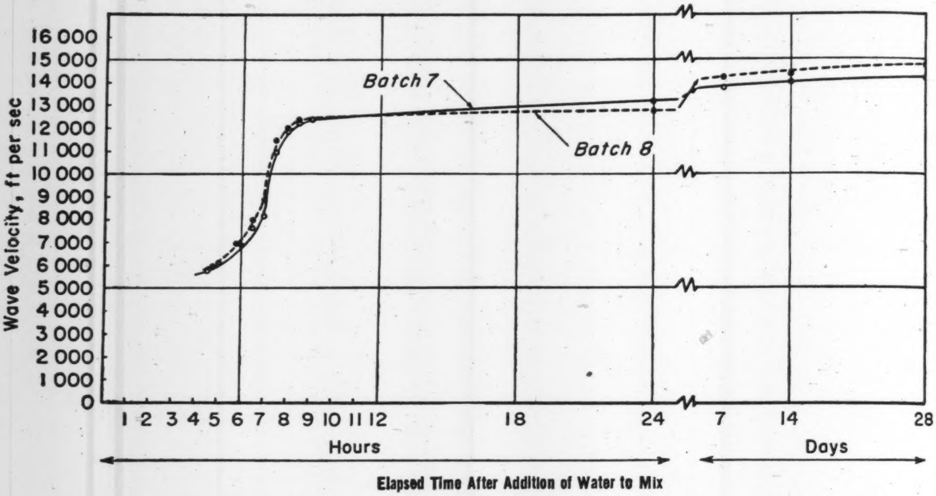

FIG. 7.—Average Wave Velocities Through Concrete Made From Type IV Cement.

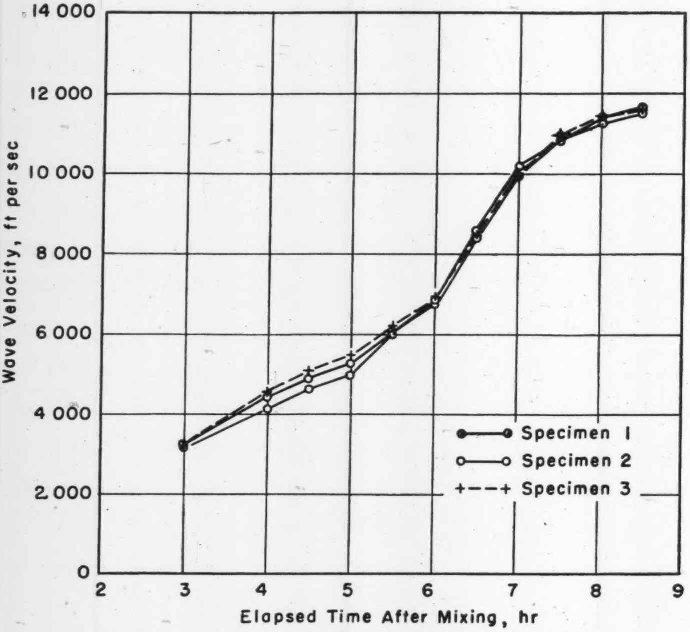

FIG. 8.—Rate of Change of Velocity (Batch 3, Mold 1).

per sec. From this initial value it increased at an accelerating rate for several hours. At the end of some period of time, the rate of change in velocity decreased sharply during a relatively brief interval. Velocities then continued to increase at a slow rate throughout the duration of the tests. Only very small differences were observed between the three companion specimens in any given mold. Figure 8 shows the results of tests on a typical group.

It seems desirable especially for the evaluation of acceleration or retarding admixtures, to designate a specific time as the time of set. From a study of the data it appeared that the time which could be most consistently reproduced was that designated by the intersection of lines drawn tangent to the curve before and after the interval during which the rate of change in velocity decreased sharply. Figure 9 shows a comparison of results of velocity tests on concretes made from the four cements used in this study. The tangents have been drawn to indicate the time of set. A comparison of the setting times so determined with the setting time of the cement alone, as determined by the Gillmore needle, is shown in Table I.

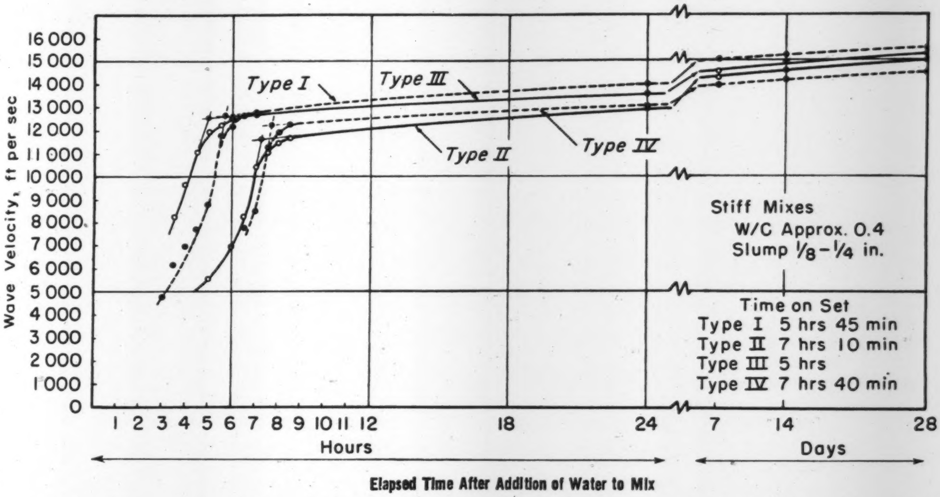

FIG. 9.—Comparison of Wave Velocities Through Concretes Made From Types I, II, III, and IV Cements, Stiff Mixes.

TABLE I.—COMPARISON OF SETTING TIME OF CONCRETE AND CEMENT.

Type Cement	Time of Set	
	Concrete	Cement
Type I	5 hr, 45 min	5 hr, 45 min
Type II	7 hr, 10 min	6 hr, 50 min
Type III	5 hr	4 hr, 15 min
Type IV	7 hr, 40 min	8 hr

It is obvious that the very stiff mixes used in the first eight batches, although convenient for early testing, were outside the range of consistencies usually found in either laboratory or field concrete. Two wetter batches were mixed with a water-cement ratio of 0.50 by weight. To minimize the slumping of the beam ends, the end plates were left on the forms for 2 hr or more after the concrete was mixed. Attempted tests on these specimens were entirely unsuccessful. Although little slumping of the ends was noted, a close examination revealed numerous small cracks opening across the top surfaces of the beams. It is felt that these were responsible for the poor test results.

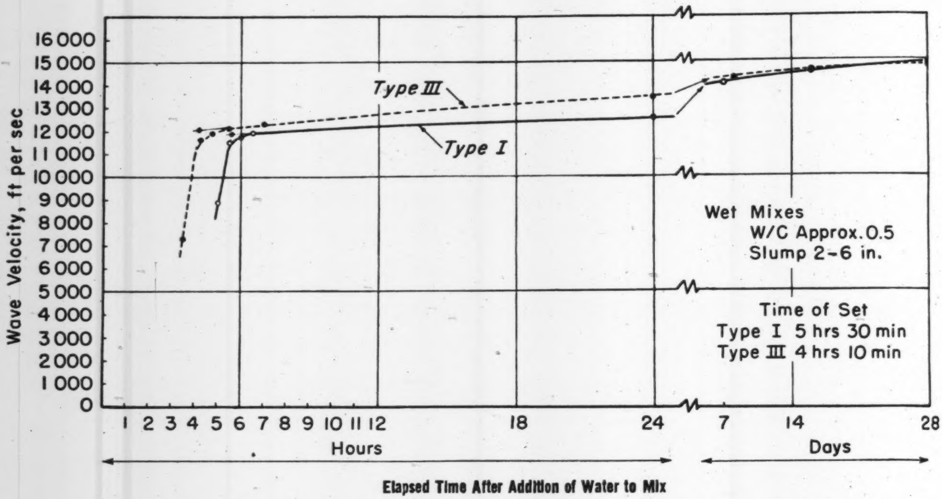

FIG. 10.—Comparison of Wave Velocities Through Concretes Made From Types I and III Cements, Wet Mixed.

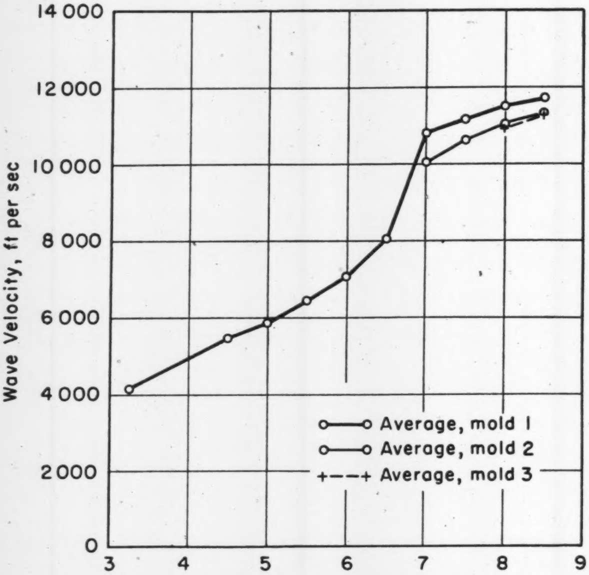

FIG. 11.—Rate of Change of Velocity (Batch 4).

Two additional batches, previously referred to as 9 and 10, were made. In the batch made from type I cement (batch 9) end plates were not removed from the forms until 5 hr after mixing; in the batch from type III (batch 10) they were removed $3\frac{1}{2}$ hr after mixing. In both cases, velocity tests were quite successful and results were similar to those obtained from the stiff mixes. Data are shown on Fig. 10. Setting times determined for these concretes were 5 hr, 30 min for that containing type I cement and 4 hr, 10 min for that made from type III.

Throughout all tests it was noted that signals received for the first 8 hr or more were very weak. Considerable care was required of the operator to insure his identification of the leading edge of the received signal. With experience, however, the test was performed with greater facility.

The tests made on beams cast in the second and third molds were not highly conclusive (Fig. 11). In most cases, during the first 24 hr, velocities measured through these beams were slightly lower than those measured through the beams tested throughout the entire setting period. On the average, at the time setting was complete, velocities were 120 ft per sec lower through those tested during only about half of the setting period (those in second mold) than through those tested throughout the entire setting period (first mold). Velocities averaged 430 ft per sec lower through those which were not tested until setting was complete (third mold). After some curing, however, by the time seven day tests were made, no consistent variations in velocity could be measured.

CONCLUSIONS

From the results of the tests reported above, it is concluded that, at least for specimens of laboratory size, the setting time of concrete may be determined by observing the rate of change of the velocity with which small vibrations are propagated through the specimen. Time of set is taken as the time at which this rate of change decreases suddenly. If velocity is measured periodically and plotted *versus* elapsed time after mixing, this time may be determined by locating the point of intersection of tangents drawn to the curve immediately prior to and after the period during which the decrease occurs.

The soniscope appears to be a suitable instrument for measuring the desired velocities.

The test is rather difficult to perform because of the very weak signals received during the setting period. With a little experience, however, an operator is able to achieve reliable and reproducible results. It should be noted that the increase in velocity is generally so great immediately prior to the setting of the concrete that the change could scarcely be missed, even if the accuracy of measurement were poor.

The method is probably no more precise than are the needle tests for setting time of cement, since the phenomenon of final set is not an instantaneous process. The method does, however, provide a means of quantitatively evaluating the setting time of concrete, and is, apparently, the only means presently available for doing so.

It is, of course, desirable to extend the method to the measurement of setting time of concrete in place. This might be accomplished by surface tests on pavements, similar to those made in Kansas. In addition, it would be advantageous to make tests on such structural members as beams, columns, and walls. This might be accomplished by the use of forms with small, externally removable plugs or sections to permit access to the concrete. Some similar device might also facilitate laboratory tests. It is hoped that some studies of this nature will be undertaken in the near future.

Acknowledgments:

The author is indebted to Professor D. W. Lewis, Purdue Univ., for advice and assistance in setting up the experimental work outlined above and for review of the manuscript.

REFERENCES

(1) A. J. Arndt, "Discussion of 'Dynamic Testing of Concrete with the Soniscope Apparatus' by W. J. Cheesman," *Proceedings*, Highway Research Board, Vol. 29, p. 176 (1949).

(2) W. J. Cheesman, "Dynamic Testing of Concrete with the Soniscope Apparatus," *Proceedings*, Highway Research Board, Vol. 29, p. 181 (1949).

(3) H. E. Chisholm, "New Type Weighted Needles for Determining the Setting Time of Magnesium Oxychloride Cements," ASTM BULLETIN, No. 154, Oct., 1948, p. 37.

(4) W. Davis, "Time of Set of Concrete," *Proceedings*, Am. Soc. Testing Mats., Vol. 21, p. 995 (1921).

(5) O. J. Glantz and L. E. Halsted, "Mechanically Determining the Time of Set of Portland Cement by Means of the Spissograph," ASTM BULLETIN, No. 170, Dec., 1950, p. 79.

(6) R. Jones, "The Non-Destructive Testing of Concrete," *Magazine of Concrete Research*, London, No. 2, June, 1949.

(7) J. R. Leslie, "The Application of Pulse Techniques to Dynamic Testing," *Proceedings*, Am. Soc. Testing Mats., Vol. 50, p. 1314 (1950).

(8) J. R. Leslie, and W. J. Cheesman, "An Ultrasonic Method of Studying Deterioration and Cracking in Concrete Structures," *Proceedings*, Am. Concrete Inst., Vol. 46, p. 17 (1950).

(9) H. E. Miller, "Discussion of 'New Type Weighted Needles for Determining the Setting Time of Magnesium Oxychloride Cements' by H. E. Chisholm," ASTM BULLETIN, No. 154, Oct., 1948, p. 39.

(10) E. A. Whitehurst, "Soniscope Tests Concrete Structures," *Journal*, Am. Concrete Inst., Feb., 1951, p. 433.

DISCUSSION

MR. W. J. CHEESMAN[1] (*presented in written form*).—The possibilities of using the soniscope apparatus to study concretes at early ages, as Whitehurst has described, has been a matter of interest to those of us working on the pulse velocity method of testing. It may be useful to present at this time a few of the observations made in somewhat similar studies by The Hydro Electric Power Commission of Ontario. These include the increase in velocity in sections of a gravity dam as well as in laboratory specimens. No attempt has been made to correlate pulse velocity with the setting time of the cement used. However, the relationship between pulse velocity and compressive strength has been examined at ages as early as 5 hr after mixing. It has been found that a good correlation exists, particularly at early ages, between compressive strength and pulse velocity. Hence, pulse velocity might be useful as an in-place measurement on concrete to determine when it has reached adequate strength for some further operation, such as stripping of the formwork.

Figure 12 shows pulse velocity plotted against age for five mixes using the same aggregates, and having the same slump, but with water-cement ratios varying from 0.817 down to 0.441. Only type I cement was used in these tests.

The specimens used were similar to Whitehurst's—ours were 4 by 4 by 20-in. beams. However, plywood molds were used, not steel, and a rubber window was fitted into the center of each side, to which the soniscope transducers could be held. In this way it was possible to start measuring the velocity through the concretes as early as 10 min after mixing. One such specimen was cast from each of six batches of the five different mixes.

The scales which have been used for our curves are those of Whitehurst.

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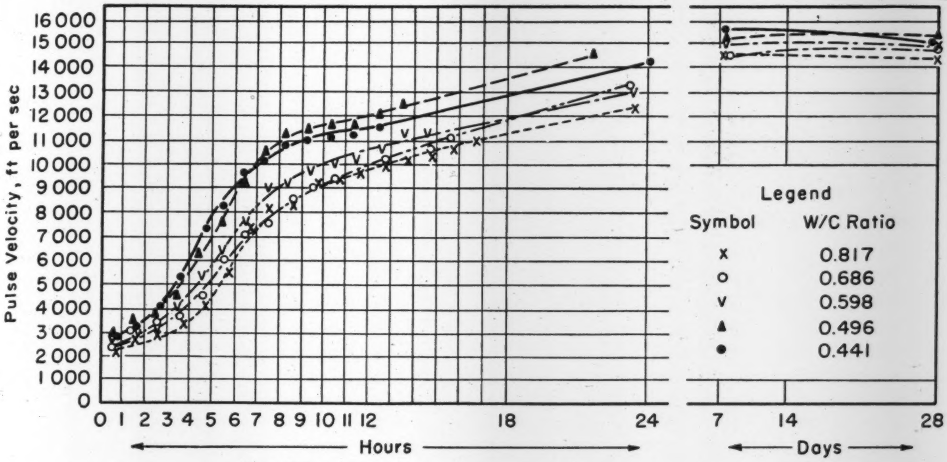
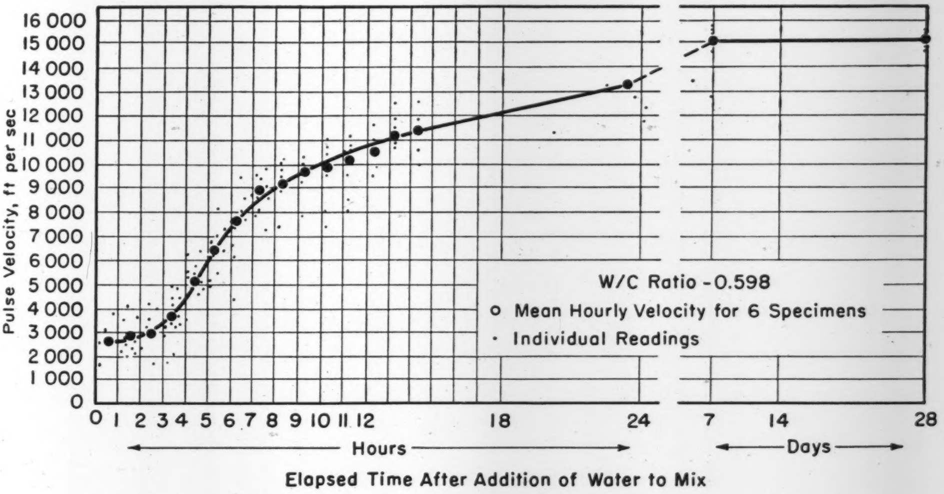
[1] Assistant Research Engineer, The Hydro-Electric Power Commission of Ontario, Toronto, Canada.



Conditions:
Cement – Type I – Vicat Initial Set – 2 hr 40 min
Vicat Final Set – 5 hr 35 min
Aggregate – Natural Sand; Crushed Stone, Max 1½ in.
Slump – 4 in.
Specimens:– 4 by 4 by 20 in.
Velocity Measured through 4 in. Path

FIG. 12.—Pulse Velocity *versus* Age—4-in. Specimens.



Condions:
Cement – Type I – Vicat Initial Set – 2 hr 40 min
Vicat Final Set – 5 hr 35 min
Aggregate – Natural Sand; Crushed Stone, Max 1½ in.
Slump – 4 in.

Specimens:– 4 by 4 by 20 in.
Velocity Measured through 4 in. Path

FIG. 13.—Pulse Velocity *versus* Age for One Mix.

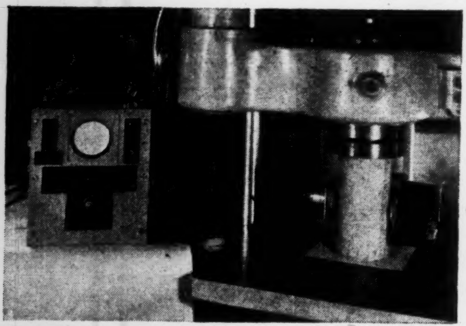
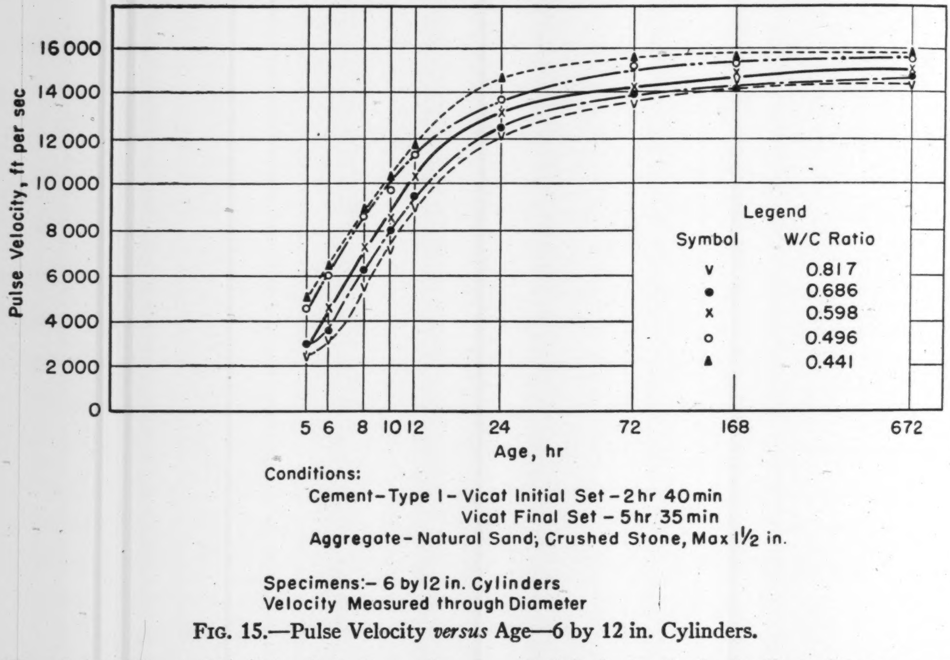


FIG. 14.—Arrangement for Taking Pulse Velocity Prior to Compression Test.



Conditions:
Cement–Type I–Vicat Initial Set–2 hr 40 min
Vicat Final Set–5 hr 35 min
Aggregate–Natural Sand; Crushed Stone, Max 1½ in.

Specimens:–6 by 12 in. Cylinders
Velocity Measured through Diameter

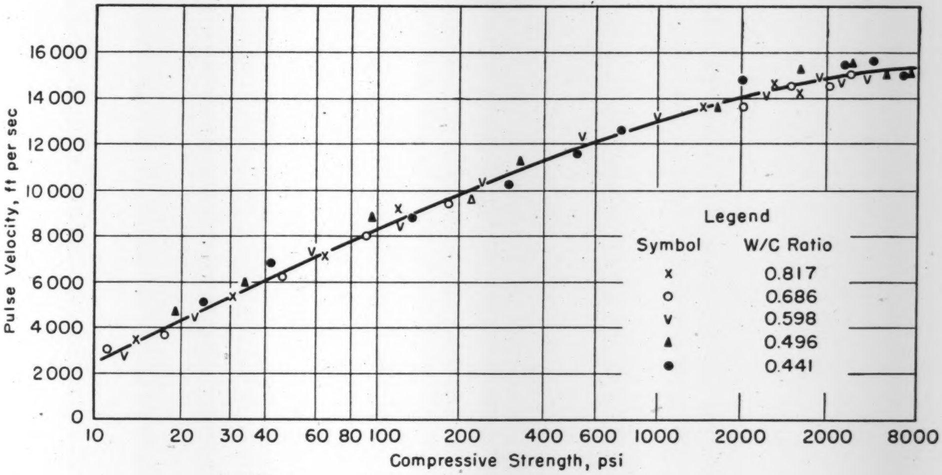
FIG. 15.—Pulse Velocity *versus* Age—6 by 12 in. Cylinders.

The first thing noticed is that the pulse velocity did not increase nearly as rapidly during the first six to eight hours, nor did it flatten out so much thereafter, as did those reported by Whitehurst. Moreover, for these specimens the curves did not stay in rank according to the water-cement ratio of the mix, but crossed and even re-crossed. It would be difficult to correlate the points of inflection of these concretes with the times of set of the cement, although they appear to be of the same order.

Figure 13 shows all of the readings taken on the six specimens of one of the mixes (water-cement ratio 0.598). The small dots are the individual test readings, while the large ones, which were used to decide the best form of the curve, are the mean hourly velocity, found by taking the average velocity and the average age of the specimens, when tested, during each hourly period. The test points show that very considerable scatter was obtained in the readings, and this is typical of all such tests we have made.

In the same series of tests in which the above prisms were made, sets of standard 6 by 12 in. compression test cylinders were cast, and tested at ages from 5 hr up to 91 days. At the very early ages it was found unwise to handle the specimens very much for fear of damaging them. Therefore, the soniscope apparatus was arranged so that the pulse velocity could be measured through the diameter of the specimens as they were in place in the compression testing machine (Fig. 14).

Six cylinders of each mix were tested at each age, and the curves of pulse velocity against age, on a logarithmic scale are shown in Fig. 15. For these specimens the velocities kept well in order of the water-cement ratios. Moreover, the spread of the six tests at each point (not shown on the graph) was considerably less than in the case of the prisms.



Conditions:
Cement – Type I – Vicat Initial Set – 2 hr 40 min
Vicat Final Set – 5 hr 35 min
Aggregate – Natural Sand; Crushed Stone, Max $1\frac{1}{2}$ in.
Slump – 4 in.

Specimens: 6 by 12 in. Cylinders
Velocity Measured through Diameter

FIG. 16.—Pulse Velocity *versus* Compressive Strength—6 by 12 in. Cylinders.

Figure 16 shows the pulse velocity plotted against the compressive strength of the cylinders at the various ages: 5, 6, 8, 10, and 12 hr, 1, 3, 7, 28, and 91 days. Using a logarithmic scale for the compressive strength gives a nearly straight line over the greater part of the range studied, and the one line appears to be equally applicable to the full range of water-cement ratios covered. Using the same aggregate, other variations in the mix, such as the use of type III and type I cement with admixtures can as fly ash and $CaCl_2$, and air-entrainment, have been tested. All followed the same velocity-strength line except the air entrained mix, for which velocities were slightly lower for corresponding values of strength.

Close examination of this curve shows that the strength discrimination of the pulse velocity test is not great at the higher strength values. It is for this reason that only by starting the tests at the early ages was it possible to find the general shape of the velocity-strength relationship.

This poor strength correlation in the range of strength most usually under consideration (that is, above about 2000 psi) is a feature believed to be common to all tests which are a measure of the Young's modulus of concrete. However, the pulse velocity appears to afford a satisfactory method of gaging the early-age strength of concrete, and the method has the advantage that it can, in most cases, be used on the concrete in place in the work.

An example of how this has been done is a case in which it was desired to follow the increase in strength in two similar sections of a gravity dam. One section was made with plain concrete, and the other contained an admixture. These test sections were relatively small, being 12 ft high, 36 ft long, and 14 ft thick; volume about 250 cu yd. Five cut-out arrangements in the formwork were provided in opposite locations on the upstream and downstream faces of the sections. These cut-outs were removed as soon as the concrete had set up adequately and pulse velocity measurements made through the sections. Because of the high attenuation of the sound pulses in plastic concrete, pulse transmissions were not achieved through the 14-ft path until the concrete was about 24 hr old. From then on it was possible to compare the build-up of strength of the concrete in place in the two sections (Fig. 17).

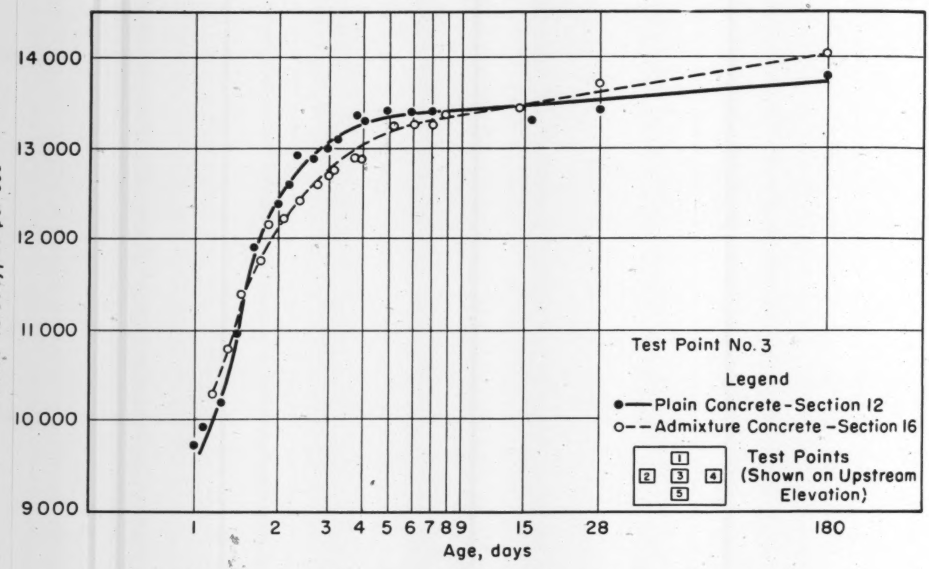


Fig. 17.—Pulse Velocity *versus* Age for Plain Admixture Concrete.

Mr. W. H. Price[2].—The lowest velocity shown by Mr. Cheesman (Figs. 12 to 16) is about 3000 ft per sec, with a strength of the concrete shown for that velocity. At what age was that concrete tested, and was its strength measured?

Mr. Cheesman.—The earliest age at which we have comparisons of pulse velocity and compressive strength is 5 hr from mixing.

Mr. Price.—Apparently Whitehurst and Cheesman did not make measurements on plastic concrete. In tests made in the Bureau of Reclamation Laboratory, velocities as low as 400 ft per sec were obtained through plastic concrete having a 3-in. slump with an interval timer. In this interval timer, a calibrated condenser is charged during the time the pulse travels between two pick-ups. The velocity of 400 ft per sec is so low that its accuracy is questioned. This was checked by removing the concrete and filling the mold, a 1-ft cube, with water. A velocity of 4780 ft per sec was obtained through the water. When some of the water was replaced with $\frac{3}{4}$-in. stone, a velocity of 2020 ft per sec was obtained and when some of the water was replaced with No. 4 size stone a velocity of 610 ft per sec was obtained. The velocity of sound through rubber is only about 177 ft per sec, so it is not impossible to obtain very low velocities through certain materials. We intend to check our results further with the idea of using this interval timer method to measure the time of set of concrete.

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[2] Materials Engineer, Bureau of Reclamation, Denver, Colo.

MR. E. A. WHITEHURST (*author*).—Mr. Cheesman does not have any strength relations down there but he has velocity readings at early ages. What were the earliest velocity readings?

MR. CHEESMAN.—The earliest velocities were taken 15 min after mixing. The lowest read was 1500 ft per sec, which is just slightly more than the velocity of sound in air. We have not made any tests aimed at seeing how low velocities will go in plastic concrete.

MR. WHITEHURST.—I should like to suggest a possible reason for Mr. Price's very low readings. I had occasion to observe the operation of the interval timer for several weeks during 1949. As Mr. Price has indicated, the reading is made on a voltmeter, and no arrangement is made for visual observation of the test signals. It was observed that under some conditions the measured transit time through a given concrete path would vary by ±10 per cent as the force of the hammer blow was varied. Apparently this was due to the fact that while a very sharp energy front reached the first pick-up to begin the timing process, passage through the concrete to the second pick-up resulted in considerable attenuation of this energy front. It could then be visualized as a sloping rather than a vertical wave, and the level necessary to trigger the pick-up would occur some time after the point of origin of the disturbance.

During the tests reported here, we found that the attenuation of a wave passing through plastic concrete was much greater than that of a similar wave passing through older, hardened concrete. I suspect that this may have influenced Mr. Price's tests and made his measured time intervals too great. I believe that it would not be too difficult to photograph the signals at both of the interval timer pick-ups. It might be very interesting to compare the shapes of the waves at the two points and see if some correction should not be applied to the measured transit times.

MR. R. E. DAVIS.[3]—I was seeking an explanation of low velocities, because the wave velocity in water is 4500 to 5000 ft, and I can see no way in which the velocity in fresh mortar or concrete could be less than that in water, unless air was present. Were these air-entrained concretes?

MR. CHEESMAN.—No. That matter of minimum velocity is a point that has been given quite a bit of consideration. Some Danish investigators, I believe, have worked out mathematical equations for the increase in velocity in concrete, assuming that the initial velocity is that in water.

One thing we did was to take a container of water; measured the pulse velocity through that and then dropped a handful of sand into it. The attenuation went upward and the velocity apparently went down considerably, down to the order of 1000 to 2000 ft per sec. As the sand settled to the bottom, the velocity came back up to the velocity of the water.

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[3] Professor of Civil Engineering, and Director, Engineering Materials Lab., University of California, Berkeley, Calif.

MR. S. B. HELMS.[4]—Two years ago we studied the development of tensile strength in pebble concrete mortar using types III and IIIA cements. I am reminded of our tests because Mr. Whitehurst shows a steep increase in velocity which is remarkably similar to the sudden development of tensile strength values which we found.

A small beam mold was used which enabled us to test a 2 by 2-in. cross-section in tension from the time when the sides could be removed from the center section of the mold, through the period where strength was too high for us to measure with our device. The approximate range of 0.5 to 25 psi was covered and we did not go beyond that.

Our tensile strength results did not extend into the region beyond the point of inflection noted where the soniscope curves are shown to flatten out. According to my concept the tensile strength would continue to rise steeply during the interval of 6 hr to 24 hr elapsed time, but we have only cantilever test data and cursory standard briquet test data which were obtained for that time region.

MR. F. E. RICHART.[5]—I was surprised Mr. Whitehurst made no mention of the sonic modulus of elasticity as related to this new apparatus. I assume that the apparatus would be adapted to the determination of modulus of elasticity.

MR. WHITEHURST.—Over a period of several years we have made velocity tests on some hundreds of prisms and quite a few cylinders for the purpose of computing the dynamic modulus of elasticity and comparing it with the modulus determined by the flexural resonance or so-called "sonic" test. In perhaps 90 per cent of the cases, agreement between the two methods has been good. In the other 10 per cent, however, the differences have been sufficiently great to cause us to wonder whether or not something may have been overlooked in applying the theoretical formulas to a material such as concrete.

I think this raises the question of just what is gained by making this calculation. To my knowledge, the dynamic modulus of elasticity of concrete, per se, has very little use. It is used primarily as a relative measure of concrete durability, usually to evaluate changes occurring during some period of natural or artificial weathering. If we are to compute this modulus from a velocity test we must accept a formula, concerning which there still seems to be some argument. We must assume a value of Poisson's ratio and, at least for concrete in place, we must assume a value for density. Having made all of these assumptions, we arrive at a figure which is used in comparison with similarly derived figures to define a durability record over a period of time. The velocity, which may be very accurately measured without assumptions of any nature, appears to be an equally good relative measure of concrete durability.

On the basis of these considerations, we are presently inclined to record only the velocity, calculating the dynamic modulus on rather infrequent occasions.

MR. P. H. KAAR.[6]—I should like to comment on Mr. Helms' remarks regarding tensile strength of concrete. Similar tests of lightweight aggregate concrete were made at The Fritz Engineering Laboratory, Lehigh University. The very early strength specimens were cast in reduced section tension test forms. They were tested in the form by dead weights. This method eliminates the handicap of handling fresh concrete specimens as they do not have to be moved from the form for test.

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[4] Research Engineer, Lehigh Portland Cement Co., Allentown, Pa.

[5] (Deceased), University of Illinois, Urbana, Ill.

[6] Civil Engineer, U. S. Bureau of Mines, Bethlehem, Pa.

The strength curves were similar to those presented here. I mention it only to indicate that, if you do not have the apparatus previously mentioned, you can still perform these tests.

MR. BRYANT MATHER.[7]—Mr. Whitehurst mentioned the desirability or necessity of placing the transducers directly in contact with the concrete. Mr. Cheesman mentioned the use of rubber windows. Does Mr. Whitehurst believe in the use of something between the transducer and the concrete?

MR. WHITEHURST.—There is no objection to something between the transducer and the concrete so long as a very intimate bond can be effected on both sides of the intermediate material. We have worked with an inch or more of water, for instance, between the transducer and the concrete, with very good results.

I understand from Mr. Cheesman that he slits the top of the rubber window and introduces a little water behind your rubber window?

MR. CHEESMAN.—That is true.

MR. WHITEHURST.—I am quite sure that, as long as you can keep a sufficiently intimate bond to pass this minute vibration, there is no objection to using such a device.

MR. F. N. WRAY.[8]—Mr. Cheesman mentioned the possible application of this impulse velocity test for the study of behavior of concrete in structures—during the early hardening period, I suppose. I should like to ask, is it applicable for reinforced concrete members and reinforced-concrete pavement?

MR. CHEESMAN.—Mr. Whitehurst and I have both had experience in measuring velocity in reinforced concrete. Speaking from my own experience, there is some influence of the reinforcing if the pulse velocity shots are taken within 2 in. of and in, a direction parallel to the reinforcing. That is, if there is a very tight pattern of reinforcing, there will be trouble. If you have an open pattern, one can get readings in between the reinforcing.

MR. WHITEHURST.—In general, I would agree with Mr. Cheesman. I have never been able to find any influence of reinforcing in, say, a pavement slab. I have on one occasion felt that velocity measurements were being influenced by reinforcing in the deck over a spillway gate in a dam which was very heavily reinforced. Outside of that, I know of only one occasion on which we had this influence, and we found it was a steel member with a little concrete around it.

(*author's closure*).—The several comments received since this paper was presented indicate an increased interest in the velocity approach to dynamic testing of concrete. As suggested by some of these comments, the author agrees that the time of set of concrete, in itself, is probably of little importance. The measurement of it, however, assumes somewhat greater importance in view of the efforts of the ASTM and other organizations to evaluate the various admixtures which are being suggested as accelerators or retarders. The author feels that the method described in this paper is one method of making such an evaluation. Mr. Cheesman's data (Fig. 17) would appear to verify this opinion. It is to be hoped that future efforts will result in simplification of the apparatus and the technique to a stage at which it will be much more useful.

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[7] Engineer (Concrete Research), Concrete Research Div., Waterways Experiment Station, Jackson, Miss.

[8] Engineer of Design, Highway Research Board, Washington, D. C.

METHOD OF MAKING VIBRATED DRY-TAMP CONCRETE CYLINDERS APPLIED TO TESTS OF LIGHTWEIGHT AGGREGATE AND BLOCK MIXTURES*

BY S. B. HELMS[1] AND A. L. BOWMAN[1]

SYNOPSIS

The Vibratamp apparatus was developed for use in laboratory investigation of lightweight aggregates where standardized specimens are required. Hand-tamped units, because of particle fracture and overdensification, are of questionable value in comparisons where reproducibility is essential.

Vibrated dry-tamp cylinders were made for three test programs in which the significance of the results is dependent upon standardized fabrication of specimens.

In test series No. 1, the effect of varied amounts of No. 100 sieve dust in lightweight aggregate was studied using four typical materials including expanded slag and expanded shale. With test concentrations fixed at 5, 15, and 25 per cent by weight, optimum properties were found at 15 per cent dust.

In test series No. 2, Types I, IA, III, and IIIA cements were used with an expanded slag mixture at 24 blocks per sack yield. The comparisons showed that water requirement and density were practically unaffected and that no significant strength or absorption differences occurred when respective air entraining cements were used.

In series No. 3 the same cements were used at 24 and 30 block per sack yields with sand and gravel aggregates. The results of freezing-and-thawing tests show a significant improvement due to air-entraining cement.

There is a definite need for a convenient method of simulating concrete-block densification in the laboratory for purposes of investigating and evaluating lightweight aggregates in this type of dry-tamped concrete mixture, particularly when only limited quantities of material are available. Our first attempts to make specimens of this kind about four years ago, were stimulated by numerous problems that had required the preparation of hand-tamped specimens. Such mixtures had been placed in regular 3 by 6-in. steel molds by hand compaction using flat-end tamping rods which tended to fracture coarse lightweight aggregate particles depending on the vigor of the operator. Initial trials with a crude-looking device showed that cylinders could be compacted readily by vibration, pushed out of the mold, and carried to the moist room on glass plates. H. T. Williams of Duquesne Slag Products Co. had made 4 by 8-in. vibrated cylinders of this kind with success and disclosed the details of his test method and apparatus.

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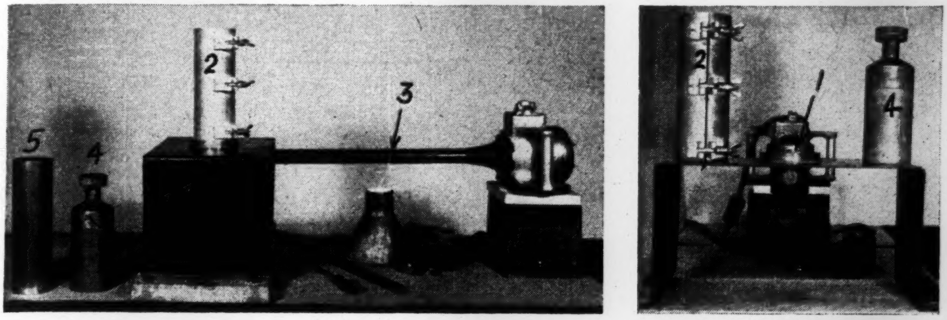
* Presented at the Fifty-Fourth Annual Meeting of the Society, June 18–22, 1951.

[1] Research Engineer and Research Chemist, respectively, Lehigh Portland Cement Co., Allentown, Pa.

In 1940, G. W. Hutchinson reported in the *Journal* of the Am. Concrete Inst. use of the vibrator described in a paper in the August, 1939, ASTM BULLETIN disclosing an adaptation of a spud vibrator that was used to place concrete in 6 by 12-in. molds; it was rigged to consolidate several specimens simultaneously. Although the objective was somewhat different from ours the method is mentioned because the same kind of vibrator was used.



(*a*) (*b*)

FIG. 1.—Vibratamp Apparatus.

1. Steel 3 in. diam. pedestal supported on a 9 by 15-in. vibrating table made from sheet steel 0.115 in. thick. The pedestal is held in place by a threaded chuck which acts as a retainer.
2. Steel 3 by 9-in. cylinder mold.
3. Viber spud vibrator (Model No. 1-9000 rpm).
4. Steel plunger weighing 12.9 lb (2.94 by 8.4 in.).
5. Pushout pedestal and other accessories—rodding and scarifying tools, funnel, motor support.

The vibrating table and pushout pedestal are bolted to the steel laboratory bench.

APPARATUS AND PROCEDURE

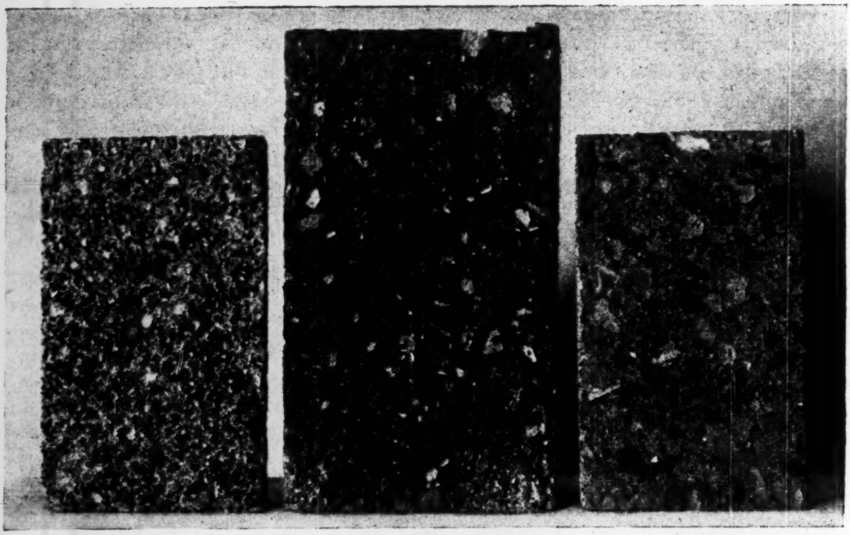
The device used in our earliest trials was subsequently improved in appearance but was practically the same as the final arrangement shown in Fig. 1(*a*). The only difference is that the present cylinder mold is taller and permits the fabrication of full height 3 by 6-in. cylinders whereas the early specimens were limited to 5.5 in.

Figure 1(*b*) is an end view of the vibratamp device which shows how the spud is fastened in the chuck. It is held in place by two Allen screws. The motor drive of the vibrator rests on sponge rubber and $\frac{3}{8}$ in. thick soft rubber sheets are employed as isolators between the vibrating table and the wooden mounting.

In the fabrication of 3 by 6-in. cylinders, aggregates are presoaked for 30 min to reduce absorption and are then mixed with cement either in a small Lancaster mixer, or by hand. Water is added until paste streaks are apparent in the trowel test of packed-down concrete. An oiled disk pallet is placed on the pedestal and the mold is tightened in place. A trial cylinder will determine the amount of batch needed and show whether the water addition is right.

Specimens are compacted stepwise in three layers, approximately one third of the weighed charge being scooped into the mold each time. Each lift is lightly rodded about 13 times with a $\frac{5}{8}$-in. bullet-pointed rod before vibrating for a short period under the weight of the plunger.

The original vibration intervals of 15 sec for the first two layers and 30 sec for the top lift were later reduced to 10, 10, and 20 sec. A good knit between layers is produced by scarifying the surfaces smoothed by the plunger in the vibration steps.



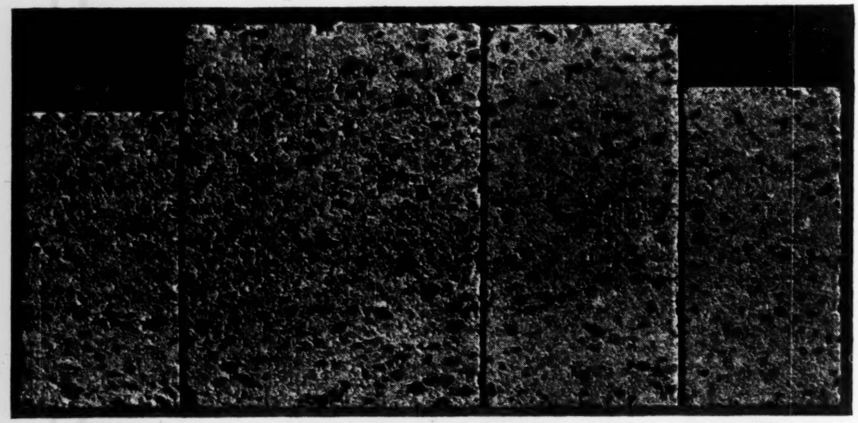
(*a*) (*b*) (*c*)

FIG. 2.—Sawed Sections Comparing Vibratamp Units with Cinder Block.

(*a*) Sintered Clay Aggregate 3/8 in. to dust, fineness modulus 4.25, yield 24 blocks per sack. Fresh density 84 lb per cu ft.
(*b*) Section Cut from Solid 4 by 8 by 16 in. Cinder Block—Besser Machine.
(*c*) Expanded slate aggregate 1/2 in. to dust, fineness modulus 4.10, yield 24 blocks per sack. Fresh density 101 lb per cu ft.

This view of internal structures shows that the laboratory specimens are reasonably similar to the type of block unit produced by commercial block-making machinery. However, despite this illustration, special trials were made to get a more specific comparison. The vibratamp apparatus was taken to a block plant so that specimens could be made from regular production batches and compared with the blocks being manufactured. Specimens were made from a mix of fixed proportions containing Type III cement, expanded slag, fine sand, and an air entraining admixture.

Figure 3 compares the internal structure of specimens made by the laboratory method and by a Besser machine. The descriptive physical data show that the similarity is not limited to appearance only.



(*a*) (*b*) (*c*) (*d*)

FIG. 3.—Sawed Sections Comparing Test Cylinders with Manufactured Blocks.

	(*a*)	(*b*)	(*c*)	(*d*)
Specimen	3 by 6-in. cylinder	6-in. block	4-in. block	3 by 6-in. cylinder
Saturated Density, lb per cu ft	93.3	93.3	92.9	93.2
Absorption, lb per cu ft	13.4	14.8	12.6	12.2
Vibration Interval, sec	15, 15, 30	11[a]	11[a]	5, 5, 10

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[a] Total cycle was 17 sec.

Specimens (*a*) and (*b*) represent a coarsely graded batch while (*c*) and (*d*) illustrate a batch relatively high in fines. Complete physical data were obtained on these specimens which, like the photograph, show definite similarity and demonstrate that vibratamp specimens can be representative of the type of concrete that has been molded in commerical block making machinery. It was found in the case of the finer mix that the only change in technique needed to get a rather close reproduction was to reduce the vibration time interval to lessen densification and bring about a commensurate reduction of strength.

After compaction is completed, the split-tube mold is loosened and stripped using the pushout pedestal. Specimens are then transferred from the disk pallet to a small glass plate and carried to the curing room.

It has been observed that optimum compaction is obtained when there is enough water in the mix to cause slight leakage of paste from the bottom of the mold during consolidation.

TABLE I.—GRADINGS AND DENSITY OF COMBINED LIGHTWEIGHT AGGREGATES AS USED IN TEST SERIES NO. 1.

Aggregate	Percentage Fines[a]	Total Per cent Retained, Weight Basis[c]							Fineness Modulus	Dry-Loose, lb per cu ft[d]	
		⅜ in.	No. 4	No. 8	No. 16	No. 30	No. 50	No. 100		⅜ in. to No. 8	Passing No. 8 Sieve
Slag A	5	0	28	39	55	70	84	97	3.73	no test	no test
	15	0	25	35	50	65	78	90	3.43	no test	[b]
	25	0	24	34	47	60	72	83	3.20	no test	no test
Slag B	5	0	20	35	52	68	82	97	3.54	44.7	61.0
	15	0	20	35	50	64	77	90	3.36	44.7	66.2
Slate C	5	0	24	42	56	70	84	97	3.73	35.1	47.5
	15	0	23	41	54	66	79	91	3.54	35.1	50.7
	25	0	22	39	51	62	74	85	3.33	35.1	54.3
Shale D	5	0	26	45	60	74	86	97	3.88	50.0	60.6
	15	0	25	44	57	71	81	92	3.70	50.0	63.8
	25	0	24	43	55	67	76	86	3.51	50.0	67.2

[a] The percentage of portion passing No. 8 sieve which passed No. 100 sieve.

[b] A tested sample fineness modulus 2.55 and 9 per cent passing No. 100 sieve had a loose density of 67.5 lb per cu ft.

[c] Wet sieve tests of fractions passing No. 100 sieve:

	Slag A	Slag B	Slate C	Shale D
Per cent passing No. 200 sieve	67	70	71	77
Per cent passing No. 325 sieve	51	47	54	61

[d] Loose density of various fractions of lightweight aggregate, lb per cu ft.

Aggregate	⅜ in. to No. 4	No. 4 to No. 8	No. 8 to No. 30	No. 30 to No. 100	No. 100 to Pan
Slag B	43.5	48.5	52.4	55.6	56.8
Slate C	31.4	36.8	34.6	43.5	55.9
Shale D	54.3	53.2	54.2	53.1	60.3

The resultant structure is largely dependent upon the grading of the aggregate. Figures 2 and 3 illustrate this point and to show that either dense matrices or open texture units are attainable when the aggregate is graded accordingly.

VIBRATAMP METHOD APPLIED TO TESTS

The new method has been utilized to produce the basic laboratory specimens in connection with three test projects.

Test Series No. 1:

The first series determined the effect of varied amounts of 100 mesh dust on density and strength of lightweight dry-tamp concrete. In order to test the aggregates in identical gradings, a loose-volume proportioning system was adopted whereby ⅜ in. to No. 8 coarse aggregates were made by combining 3 volumes of ⅜ in. to No. 4 with 2 volumes of the No. 4 to No. 8 sieve size and fine aggregates were prepared by combining 5 volumes Nos. 8 to 30, and 4 volumes of No. 30 to No. 100 sieve fractions with three different amounts of 100-mesh fines which were fixed at 5, 15, and 25 per cent by weight. The actual combined gradings have been computed for reference and are given in Table I. The column of per cent fines defines the various aggregate samples, and the mix designations in Table II refer to these dust concentrations in order to distinguish between fine aggregates made with a given material.

Preparation of Specimens.—Equal volumes of coarse and fine aggregate were weighed dry in five separate fractions and after presoaking were mixed with Type I cement in a small Lancaster mixer for 5 min. Water was added to reach the proper consistency as judged by the trowel test. The eight 3 by 5.5-in. cylinders for each mix were derived from two batches and subdivided for test at 7 and 28 days. Specimens were moist cured 7 days and those for the 28-day test were transferred to storage at 50 per cent relative humidity until broken.

Records of height and weight of specimens were kept which permitted the computation of yield and air dry unit weight. The units shaped by the original mold were 3 in. in diameter and the new mold, shown in Fig. 1(*a*), made 2.99-in.-diam cylinders in tests subsequent to Series No. 1; computations assumed an average cross-section of 7.07 and 7.02 sq in. respectively.

TABLE II.—COMPOSITION AND PROPERTIES OF DRY-TAMPED LIGHTWEIGHT CONCRETES (3- BY 5.5-IN. CYLINDERS).

Mix[a]		Quantities per cubic foot (Compacted), lb					Yield		Air Dry Basis		Compressive Strength (Average of 4 Test Results), psi	
		Type I Cement	Water	Fine Aggregate Passing No. 8[b]	Coarse Aggregate Retained on No. 8	Total	cu ft per sack	Blocks per sack[c]	Weight Loss, per cent[d]	Density, lb per cu ft	7 days Moist	28 days Air-Dry[d]
Expanded Slag A	A5	11.4	11.9	48.2	27.6	99.1	8.25	24.8	no data	no data	776	1585
	A15	11.9	12.8	53.8	28.7	107.2	7.92	23.8	no data	no data	1160	2355
	A25	11.6	12.1	55.7	28.1	107.5	8.10	24.3	no data	no data	1560	2840
Expanded Slag B	B5	11.5	14.8	49.6	26.7	102.6	8.16	24.5	8.0	94.4	...	1400[e]
	B15	11.6	14.1	51.6	27.8	105.1	8.11	24.3	7.1	97.6	...	1835[e]
Expanded Slate C	C5	11.2	11.8	31.0	23.0	77.0	8.40	25.2	8.5	70.4	871[f]	1250
	C15	11.4	12.3	33.6	23.3	80.6	8.27	24.8	6.9	75.0	1250	1905
	C25	11.8	12.3	37.2	24.2	85.5	7.98	23.9	7.2	79.3	1550	2405
Expanded Shale D	D5	11.4	12.9	39.8	32.9	97.0	8.24	24.7	7.3	90.8	1620	2090
	D15	11.7	12.8	43.1	33.7	101.3	8.03	24.1	6.1	95.2	2260	3320
	D25	11.6	13.0	45.1	33.6	103.3	8.07	24.2	5.8	97.3	2440	3650

[a] Numbers denote per cent passing No. 100 sieve in fine aggregate.
[b] See Table I for grading of combined aggregate and loose densities of fine and coarse aggregates.
[c] Assumes 3 blocks per cu ft of tamped mix.
[d] Cylinders allowed to dry at 50 per cent relative humidity after 7 day damp cure.
[e] Average of 20 specimens from 4 batches, individually reported in Appendix I.
[f] Average of 3 specimens.

Results.—The results of Test Series No. 1 are presented in Table II and are summarized graphically in Fig. 4. It should be made clear that the sample of slag A had $\frac{1}{4}$ in. max size; therefore the coarsest fraction consisted of a blended material containing slag B. The practice of determining air dry density was not begun until the slag A tests were completed.

The data of Table II are of particular interest because grading and proportioning were standardized with yield closely controlled at 24 blocks per sack according to an assumed volume of 0.33 cu ft per block.

An exception to the making of eight cylinders per mix was made in the case of slag B where 20 cylinders were made from four batches on the same day. All these specimens were tested air dry at 28 days in order to determine reproducibility of the average test result. This statistical study is reported in detail in Appendix I. The average within-batch coefficient of variation was 3.3 per cent while the maximum deviation of batch average from the grand average was 3.0 per cent and the average deviation on the same basis was 1.6 per cent.

Conclusions.—1. Based on the values given in Table II it is concluded that the optimum dust percentage is 15 per cent by weight of the fine aggregate even though higher strengths were found with 25 per cent dust. The latter amount is excessive, making the aggregate too dusty for practical purposes. Also, with 25 per cent fine fines the stickiness of the concretes results in surface streaks which detract from appearance.

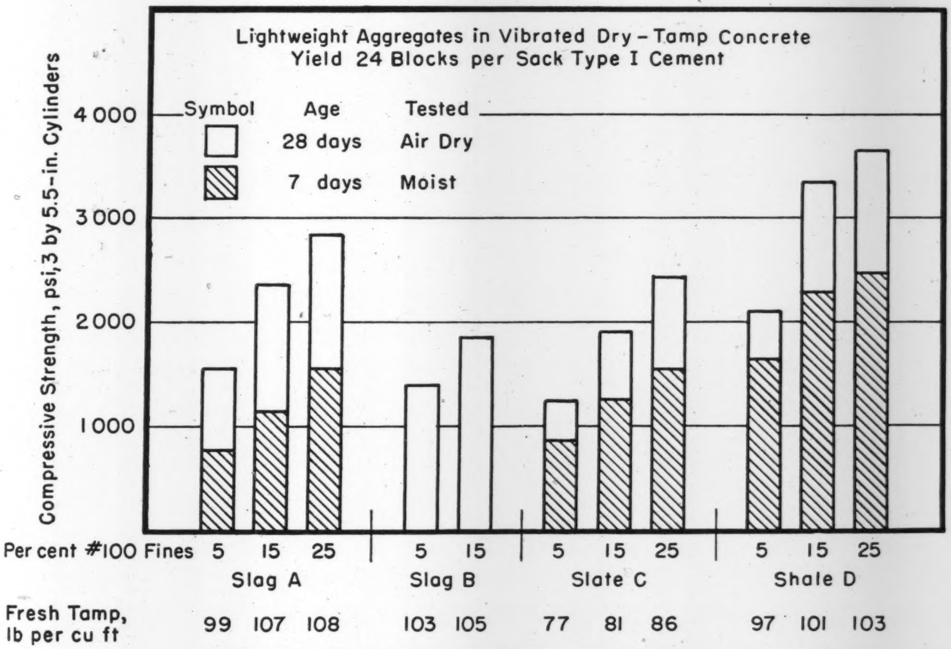


FIG. 4.—Effect of Fines on Compressive Strength.

2. Increased strength with increased fines may not be entirely due to improved densification, since substantial quantities of dusts were introduced which, because of calcining or vitrifying treatment, possess pozzolanic properties and probably raise the 28-day strength values.

3. When plotted on a strength-density basis the expanded shale and slate are found to be closely related and outstanding while the slags produce units with a lower strength characteristic.

Test Series No. 2:

In the second series, the effect of air entraining cement was studied, using a new sample of expanded slag B as the aggregate for all tests. Appendix II shows that a new supply of type I cement was used. The test conditions were the same as in the prior series except that ten cylinders were prepared for each mix from five hand-mixed batches.

Available data which specifically compare specimens molded from hand-mixed *versus* machine-mixed batches show that similar concrete strengths result from these two techniques; in two separate comparisons with different mixtures, strength was found slightly higher and slightly lower in the hand-mixed batches.

The samples of slag had been regraded to conform with combined grading B 15 of Table I. A short tabulation of sieve results shows how the fractions were changed in order to reproduce the former grading.

GRADING OF EXPANDED SLAG B AGGREGATES —SERIES NO. 2.

	Total per cent retained on									Loose Density, lb per cu ft
	3/8 in.	1/4 in.	No. 4	No. 8	No. 16	No. 30	No. 50	No. 100	No. 200	
Coarse Size (3/8 in. to No. 8):										
As received...	10	48	77	91						
As used.......	0	32	57	100						39.7
Fine Size (Passing No. 8):										
As received...			0	4	24	48	64	77	94	
As used.......				0	20	45	67	85	96	65.3

The $\frac{3}{8}$ in. to No. 8 coarse aggregate fraction had a loose density of 39.7 lb per cu ft which compared to 44.7 lb per cu ft in the earlier sample, so the particles were probably not as strong. This change evidently reduced strength compared to that for slag B in Series No. 1, but the comparisons are still indicative and have been borne out by subsequent tests. Table III lists the principal properties of dry-tamp expanded slag concretes made with plain and air-entraining Types I and III cements. It should be explained that the absorption tests were made on specimens that represented yields close to 25 blocks per sack (and 31 blocks per sack for the fifth mix), all being about 1 block per sack higher than the average yields listed. The air drying loss data are indicative of absorption values and, being more representative, show more reliably the effect of air entraining cement.

It will be noted that strengths were lower than expected and that 895 psi at 28 days for mix No. 22 in Table III compares unfavorably with 1835 psi for mix B 15 in Table II. Although a new cement lot was also used, the authors feel justified in attributing the low strength to the characteristics of the second sample of expanded slag. To support this statement, some test results are available that compare expanded slag from two sources in similar mixes made with the same type I cement.

Series No. 1 Mix Designation	Expanded Slag B[a]	Compressive Strength, lb per cu ft	
		7 Days Moist	28 Days Air Dry
B 5........	Source a (20) Source b (6)	... 638	1400 1010
B 15.......	Source a (20) Source b (6)	... 730	1835 1130

[a] Numbers in parentheses denote number of results averaged.

Because of the low apparent strength, results with slag aggregate from source b given here were not included in Table II. Rather than nullify the results of series No. 2 because of the low strength or prolong the program by testing new samples of slag B, it was decided to expand the study of air-entraining ce-

TABLE III.—COMPOSITION AND PROPERTIES OF LIGHTWEIGHT VIBRATAMP CONCRETES (3- BY 6-IN. CYLINDERS).

Mix	Type Cement	Quantities per cubic foot (Compacted), lb[a]					Yield		Air Dry Basis		24 hr Absorption, per cent	Compressive Strength, psi	
		Cement	Water	Fine Aggregate[b]	Coarse Aggregate[b]	Total	cu ft per sack	Blocks per sack	Weight Loss, per cent	Density, lb per cu ft		7 days Moist	28 days Air Dry
21......	I	11.7	16.8	48.8	23.5	100.8	8.00	24.0	11.2	89.4	16.4	600	895
22......	IA	11.7	16.7	48.6	23.4	100.4	8.05	24.2	11.0	89.4	15.4	615	1060
23......	III	11.6	16.5	48.1	23.2	99.4	8.12	24.4	9.8	89.5	14.7	850	1560
24......	IIIA	11.7	16.6	48.6	23.5	100.4	8.03	24.1	10.0	90.4	15.1	830	1400
25......	IIIA	9.4	17.4	48.5	23.4	98.7	10.0	30.0	12.0	86.9	17.2	490	905

[a] Proportions based on mix designated as B 15 in Table II.
[b] A new supply of expanded slag B characterized by lower loose density than the supply used in series No. 1.

ment by using natural sand and gravel aggregates in a third series.

Air-Entraining Cement in Expanded Slag Mixes.—The properties of the cements used are given in Appendix II. Comparisons were made using the same cements in Series No. 3 which support the validity of the relationships found using expanded slag aggregate. Therefore the results are believed to be indicative in comparing air-entraining with plain cement.

Table III shows that, in tests using expanded slag B as the basic lightweight aggregate, the use of air-entraining cement did not bring about any definite changes in water requirement, densification, compressive strength, or the absorption of vibrated dry tamp concrete.

It is known that block manufacturers do experience processing advantages when air entraining cement is used to alleviate grading deficiencies and to improve cohesiveness of freshly compacted units. These benefits would appear to be obtained without significant changes in important physical properties.

Test Series No. 3:

Freezing and Thawing Tests.—About ten years ago accelerated durability tests of commercially produced grit-concrete Dunbrick units were completed at our laboratory. Results after 400 cycles showed least weight loss and unimpaired residual compressive strength for test concretes containing admixtures that would be currently classed as air entraining materials. The compressive strength of the Dunbrick had been 4500 psi at 28 days. It appeared worth while to study the effect of air-entraining cement in grit concrete containing Delaware River aggregates and to reserve specimens from each mix for freezing-and-thawing tests. The mixes contained 55 parts of $\frac{3}{8}$ in. to No. 4 pea gravel and 45 parts of grits passing the No. 4 sieve.

PHYSICAL DATA FOR SAND AND GRAVEL AGGREGATES—SERIES NO. 3.

Aggregate	Proportion	Total per cent Retained on								Fineness Modulus	Apparent Specific Gravity	24 hr Absorption, per cent
		⅜ in.	¼ in.	No. 4	No. 8	No. 16	No. 30	No. 50	No. 100			
Pea	55	0	30	100	...	...	...	...	...	...	2.75	1.8
Grits	45	...	...	0	8	35	69	92	98	3.02	2.74	2.7
Combined	100	0	16	55	58	71	86	96	99	4.65	...	...

It was desired to evaluate frost durability on the basis of expansion as well as loss in weight; this introduced the problem of fabrication of vibratamp specimens containing gage points.

Selection of 45 per cent sand was based on trial specimens made with the Delaware River sand and round pea gravel; this proportion was found to be the optimum sand content for good compaction. In this series the vibration periods were reduced to 10, 10, and 20 sec respectively for the successive steps. Even with this shortened time, the freshly molded cylinders were compacted sufficiently and were firm when judged by applied finger pressure. Gage points were placed in the freezing-and-thawing specimens at the time of molding. To provide additional anchorage within the cylinders, each standard insert was fitted with a crosspiece of $\frac{1}{8}$-in. diameter rod $\frac{3}{4}$ in. long in a hole placed $\frac{1}{4}$ in. from the knurled end. Gage points were "vibrated" into place with some difficulty and efforts to measure expansion during freezing cycles were not altogether successful because the points became loosened and the results are of questionable significance. All specimens were cured seven days moist and thereafter for three weeks at 50 per cent relative humidity. Durability test specimens were then soaked in 70 F water for 24 hr and frozen at age 29 days. When all specimens were in readiness for unified treatment, they were subjected to regular cycles of overnight freezing under water in rubber boots, reaching a minimum

TABLE IV.—COMPOSITION AND PROPERTIES OF VIBRATAMP PEA GRAVEL CONCRETES.

Mix	Type Cement	Quantities per cubic foot (Compacted), lb					Yield		Air Dry Basis		Compressive Strength, Average of 4 test results, psi		Average Data for Two Freezing and Thawing Test Specimens			
		Cement	Water	Sand	⅜ in Round Gravel	Total	cu ft per sack	Blocks per sack	Weight Loss, per cent	Density, lb per cu ft	7 days Moist	28 days Air Dry	24-hr Absorption from Air Dry per cent[a]	21 Day Air Drying Shrinkage, per cent	Cylinder Numbers in Fig. 6	Average Weight Loss After 175 Cycles, per cent[b]
10....	I	11.4	9.0	66.0	54.0	140.4	8.23	24.7	2.9	136.4	1950	3000	3.6	0.045	1, 2	10.0
11....	IA	11.5	9.1	66.4	54.4	141.4	8.17	24.5	2.9	137.4	1995	3150	3.4	0.046	3, 4	1.6
13....	III	11.3	9.0	65.6	53.7	139.6	8.27	24.8	2.2	136.7	2920	3690	3.1	0.055	7, 8	10.5[c]
14....	IIIA	11.4	9.0	66.1	54.1	140.6	8.21	24.6	2.1	137.7	2970	3850	3.2	0.053	9, 10	3.9
15....	III	9.2	9.7	66.2	54.1	139.2	10.24	30.7	3.2	134.9	1850	2480	4.3	0.057	11, 12	15.7
16....	IIIA	9.2	9.5	66.6	54.5	139.8	10.18	30.5	3.0	135.3	1815	2600	4.0	0.054	13, 14	4.3

[a] Absorption on basis of the air dry weight.

[b] Loss based on maximum saturated surface dry weight reached during progress of durability test.

[c] One cylinder through complete test; withdrawn cylinder showed faster breakdown, 5 per cent loss at 70 cycles.

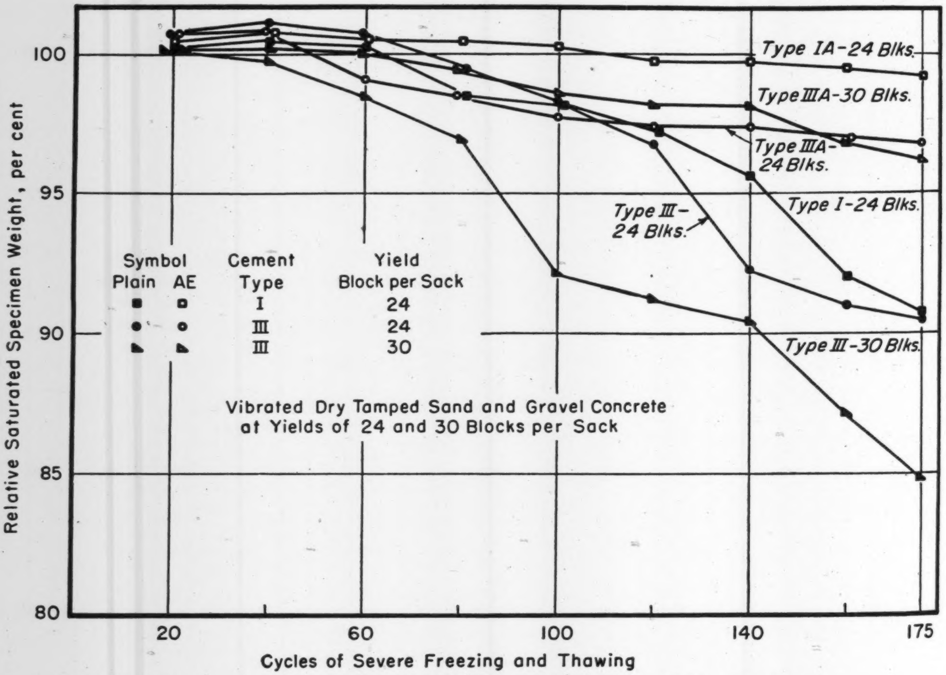
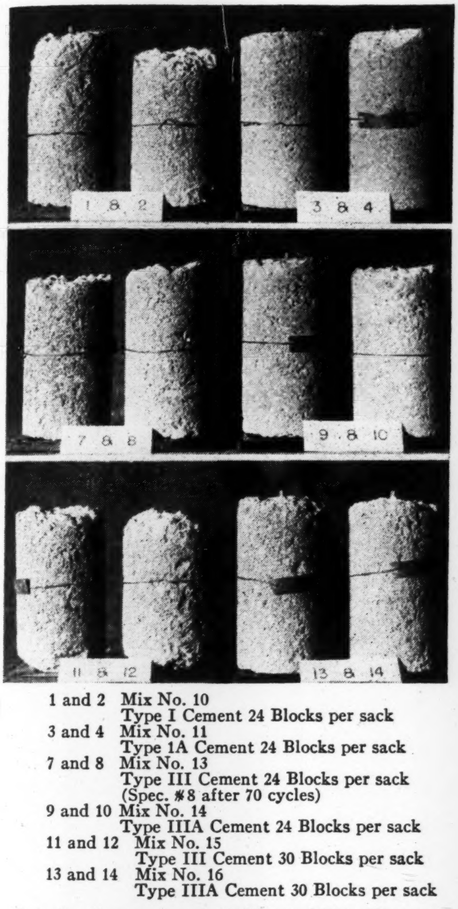


FIG. 5.—Weight Loss Record of Vibratamp Concrete Durability Specimens.

temperature of 0 F; each day they were thawed in 70 F water for a 2 to 3-hr period.

Saturated weight and length measurements were made every ten cycles at an equilibrium temperature of 70 F. Except for No. 8 all specimens were subjected to 175 freezing cycles.



1 and 2	Mix No. 10 Type I Cement 24 Blocks per sack
3 and 4	Mix No. 11 Type 1A Cement 24 Blocks per sack
7 and 8	Mix No. 13 Type III Cement 24 Blocks per sack (Spec. #8 after 70 cycles)
9 and 10	Mix No. 14 Type IIIA Cement 24 Blocks per sack
11 and 12	Mix No. 15 Type III Cement 30 Blocks per sack
13 and 14	Mix No. 16 Type IIIA Cement 30 Blocks per sack

FIG. 6.—Appearance of Vibrated Dry-Tamped Sand and Gravel Units After Exposure to 175 Cycles of Freezing and Thawing.

Results.—In the first four mixes, yields of 24 blocks were desired, and in the latter two, containing high-early strength cement, yields of 30 blocks per sack. As previously mentioned, the results in Table IV confirm the general findings of the tests of effect of air-entraining cement in expanded slag mixtures, but it will be noted that consistent small increases in density and 28-day air-dry compressive strength were found when the air-entraining cement was used.

Available data for expansion after 100 cycles showed poor correlation with weight loss data. Weight losses are listed in Table IV for all mixes after 175 freezing-and-thawing cycles were completed. In addition, the average weight loss record of the test units is plotted in Fig. 5. The condition of the specimens after exposure is shown in Fig. 6. In comparisons made at 130 cycles, specimens 1 and 2, representing Type I cement, registered an expansion greater than 0.1 per cent, but the apparent expansion of all other specimens, in so far as they could be measured, was less than 0.03 per cent. As previously stated, the loosening of gage points reduced the accuracy of the expansion measurements.

Conclusions.—1. Laboratory processing characteristics using natural aggregates were about the same for air-entraining and non air-entraining cements. The water requirements recorded in Table IV show little difference between the two kinds of cements in each of three comparisons.

2. The photographs and weight loss data show that in three separate comparisons the specimens made with air-entraining cement were found to be more resistant to damage by freezing. Since these specimens contained natural aggregates this improvement can be expected for both concrete block and Dunbrick units.

3. Air drying shrinkage, over the three-week air curing period, was practically unaffected by the use of air-entraining cement; average results of 0.046 per cent and 0.055 per cent were

recorded for Type I and Type III cements respectively.

CONCLUSIONS

The extensive test data demonstrate that the Vibratamp technique provides a useful laboratory test method which can be used to advantage for the following purposes:

1. Experimental quantities of lightweight aggregate can be evaluated by comparing with accepted commercial products.

2. Significant results can be obtained on a small scale in studies of aggregate grading, block mix proportions, and air-entraining admixtures. It is possible to study the relations of yield and strength *versus* ASTM cement type to find how yield can be increased with Type III cement while maintaining strength, under the given curing conditions, that is equivalent to a richer Type I cement mix.

3. Densification of the compacted cylinders can be varied to obtain a close reproduction of the structure of commercially produced concrete masonry units. This is shown in Fig. 3.

The vibratamp apparatus is a simple device which can be duplicated by others at moderate cost. The authors hope the method will serve others as the basis for laboratory evaluation of lightweight aggregates in dry tamp concrete.

We know of no published results of a test method treating the specific subject of laboratory scale fabrication of small cylinder units. However, a reference list is given which, in our opinion, includes articles that will be most useful to the reader.

REFERENCES

(1) M. O. Withey, "Freezing and Thawing, Permeability and Strength Tests on Vibrated Concrete Cylinders of Low Cement Content," *Journal*, Am. Concrete Inst., Vol. 6, No. 5, p. 529 (1935).

(2) C. A. Menzel, "Studies of High Pressure Steam Curing of Tamped Hollow Concrete Block," *Journal*, Am. Concrete Inst., Vol. 7, No. 1, p. 51 (1935).

(3) K. F. Wendt and P. M. Woodworth, "Tests on Concrete Masonry Units Using Tamping and Vibration Molding Methods," *Journal*, Am. Concrete Inst., Vol. 11, No. 2, p. 121 (1939).

(4) G. W. Hutchinson, "Concrete Aggregate Development on the Craytor Hydro Project," *Journal*, Am. Concrete Inst., Vol. 11, No. 3, p. 273 (1940).

(5) H. F. Gonnerman, "Vibrating Concrete in Test Cylinder Molds," Job Problems and Practice Section, *Journal*, Am. Concrete Inst., Vol. 16, No. 2, p. 134 (1944).

(6) M. O. Withey, "Considerations Involved in the Making of Freezing and Thawing Tests on Concrete," *Proceedings*, Am. Soc. Testing Mats., Vol. 46, p. 1198 (1946).

(7) PRA tests reported, "Test Waylite Concrete," *Rock Products*, Vol. 50, No. 4, p. 175 (1947).

APPENDIX I

STATISTICAL INFORMATION ON REPRODUCIBILITY OF COMPRESSIVE STRENGTH SPECIMENS.

Refer to Table I for Lightweight Aggregate Grading and Loose Density and to Table II for Composition of the Compacted Concrete Cylinders.

28 Day Air Dry Compressive Strength, psi—individual 3 by 5.5 in. test cylinders.

Mix Designation	B 5-1	B 5-2	B 5-3	B 5-4
1	1330	1340	1440	1430
2	1370	1340	1370	1430
3	1360	1570	1380	1440
4	1360	1380	1310	1440
5	1410	1460	1380	1360
Average	**1370**	**1420**	**1380**	**1420**
Coefficient of Variation, C_V	**1.9**	**6.2**	**3.0**	**2.1**

Mix Designation	**B 15-1**	**B 15-2**	**B 15-3**	**B 15-4**
1	1880	1880	1840	1990
2	1850	1730	1800	1920
3	1710	1820	1770	1850
4	1800	1780	1780	1820
5	1770	1850	1850	1880
Average	**1800**	**1840**	**1810**	**1890**
Coefficient of Variation, C_V	**3.3**	**5.1**	**1.8**	**3.1**

Average C_V 9 test groups at 7 days Table II = 3.4 min 0.6 max 6.1.
Average C_V 9 test groups at 28 days Table II = 3.8 min 0.8 max 8.3.
Average C_V 8 test groups listed above (28 days) = 3.3 min 1.8 max 6.2.

APPENDIX II

PROPERTIES OF CEMENTS USED IN TEST PROGRAMS.

Cement	No. 1	No. 2	No. 3	No. 4	No. 5
ASTM Type	I	I	IA	III	IIIA
Used in series No.	1	(Series Nos. 2 and 3)			
Chemical Constituents, per cent:					
SiO_2	22.1	21.3	20.7	20.0	20.9
Fe_2O_3	2.3	2.4	2.5	2.1	1.9
Al_2O_3	5.9	6.2	6.7	5.8	5.5
CaO	63.6	62.9	63.4	63.0	64.5
MgO	3.2	3.1	2.8	4.0	2.8
SO_3	1.8	2.3	2.3	3.0	2.8
Ignition loss	0.9	1.1	1.0	1.3	1.4
Total	99.8	99.3	99.4	99.2	99.8
C_3S	43	42	46	53	56
C_2S	31	29	25	17	18
C_3A	11.7	12.3	13.5	11.9	11.4
C_4AF	7	7	8	6	6
Physical Properties:					
Normal consistency, per cent	25.0	25.5	25.5	29.0	30.0
Air content, per cent	...	...	19.7	...	18.4
Gilmore setting time, initial	3:00	3:15	3:30	2:00	2:45
final	5:00	5:15	5:30	3:30	4:30
Fineness, per cent passing No. 325 sieve	92.0	91.4	92.5	99.6	99.7
Wagner specific surface, sq cm per g	1840	1810	1840	2870	2860
Soundness—autoclave exp., per cent	0.19	0.23	0.22	0.27	0.08
Compressive Strength, psi:					
7 day	3020	2880	2875	4960	4800
28 day	5220	4475	4100	5630	5670
Tensile Strength, psi:					
7 day	425	433	410	565	477
28 day	523	530	492	578	540

DISCUSSION

Mr. H. T. Williams (*by letter*).[1]—The results given in the paper show that the authors have set up an effective test procedure for simulating block plant mixes. No doubt many studies can be made on a laboratory scale with this type of equipment and the results reflected to block plant practice.

While this investigation has been very complete, and there is little I would suggest to modify the technique, it is evident in the tables that yields were converted to blocks per sack by arbitrarily assuming a volume of 0.333 cu ft per unit. According to dimensions, a modular 8 by 8 by 16-in. masonry unit has a solid volume of 0.5257 cu ft and core volume variations would result in units with the following volumes of concrete:

Core volume, per cent	38	40	42	44	46	48	50
Volume of unit, cu ft	0.33	0.31	0.30	0.29	0.28	0.27	0.26

I believe that many persons consider a modular unit of 45 per cent core volume a standard, although our laboratory tests of Besser units with new cores indicate core volumes of about 43 per cent. I would like to see other laboratory data on this, however.

The water requirements shown for the various mixes are apparently gross water including absorption of the aggregate since I know of no accurate means to measure absorption of lightweight aggregates, particularly the fine sizes.

The comprehensive nature of the three test series reported will make them valuable references.

Mr. S. B. Helms (*author*).—We appreciate Mr. Williams' comment referring to yield computations and have been giving this point consideration for some time. Our data are meager for the displacement of 8-in. hollow blocks, but recently we found volumes in the range of 0.28 to 0.30 cu ft which prompted us to decide to change the value to be used in future work and assume 0.30 cu ft for the average block. The tabulated yield data in the paper can be changed readily to the suggested basis which amounts to an 11 per cent increase in the number of blocks per sack.

The total water requirements as listed included water absorbed by the aggregates, but, in the case of the specimens made with sand and gravel, it is possible to derive dependable values for net water by deducting the given amounts absorbed by the natural aggregates.

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[1] Materials Engineer, Duquesne Slag Products Co., Youngstown, Ohio.

A STUDY OF WHITE COAT PLASTER BY DIFFERENTIAL THERMAL ANALYSIS*

By J. A. Murray[1] and H. C. Fischer[1]

Synopsis

White coat plasters, prepared from sixteen commercial hydrated limes, were subjected to differential thermal analysis at ages up to one year. Significant differences were found between the several hydrates, particularly in regard to such factors as the rate of carbonation of the $Ca(OH)_2$ portion of the plaster, the rate of hydration of the MgO portion, and the retention of free and adsorbed water. Evidence pointing to the delayed hydration of calcium sulfate hemihydrate was found in a few cases, while in some of the plasters indications were found of slow dehydration of gypsum at the later ages.

Positive evidence of the carbonation of the magnesium hydroxide fraction was unobtainable under the experimental conditions, although some peculiarities in the thermograms suggest this possibility. The formation of magnesium sulfate heptahydrate in dolomitic plasters subjected to wet storage conditions is believed to have occurred.

This study was undertaken as an attempt to learn what chemical changes occur in white coat plaster as time progresses.

Studies have been made (**1, 2, 3, 4, 5**)[2] but primarily with the intent of explaining plaster failures and attempting to settle the highly controversial issue of whether or not the hydration of MgO to $Mg(OH)_2$, on the wall, and the resultant expansion is responsible for these failures.

It should be emphasized that the work contained herein was not undertaken specifically for the purpose of uncovering the mechanism responsible for plaster failures but rather as a general study with the hope that some of its findings might possibly shed some light on the failure problem in one way or another.

Even if no white coat plaster failures occurred, an insight into just what occurs in the white coat on a plastered wall would be of sufficient interest to warrant an investigation. Of particular interest is the rate of carbonation of the calcium hydroxide fraction upon exposure of the white coat to normal atmospheric conditions. Free $Ca(OH)_2$ is known to be a deleterious substance when in contact with paint films or wallpaper because of its saponifying and bleaching action. It is for this reason that a freshly plastered wall should not be painted or papered immediately. At the end of several months the calcium hydroxide has generally been converted to calcium carbonate by atmospheric carbon dioxide, and, since the carbonate is an inert material with respect to the paint and paper, these materials may then be applied with a reasonable degree of safety.

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* Presented at the Fifty-fourth Annual Meeting of the Society, June 18–22, 1951.

[1] Associate Professor and Research Associate, respectively, Massachusetts Institute of Technology, Cambridge, Mass.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1212.

METHODS AND MATERIALS

Equipment and Methods:

The equipment used in this investigation consisted of a nickel crucible mounted in a vertical furnace, chromel-alumel thermocouples to measure the furnace temperature and the differential temperature between a thermally inert material (Al_2O_3) and the active sample—both carried in the nickel crucible—and two automatic recorders to record the furnace and differential temperatures. A uniform rate of heating was maintained by a motor-driven variac.

The thermograms were originally produced as two separate curves. A Leeds & Northrup Speedomax electronic recorder drew a continuous curve of differential millivoltage plotted against time, while a Leeds & Northrup Micromax electronic recorder plotted a curve of temperature (furnace) against time. These recorders were synchronized so that the chart speed of one was the same as that of the other.

For the majority of the analyses, a heating rate of approximately 10 C per min was maintained over the temperature range 400 to 1100 C. The rate from room temperature to 400 C was always slightly lower due to the inherent characteristics of the equipment.

The analysis of white coat plasters by differential thermal methods is relatively simple since all of the major constituents—gypsum, the hydroxides and carbonates of calcium and magnesium—decompose on heating with the absorption of fairly large amounts of heat. With the exception of magnesium carbonate, the presence or absence of any of these constituents is readily recognizable in the thermogram.

Each reaction has a fairly definite temperature at which the temperature differential is noticeable. This temperature is characteristic of the reacting material and serves to differentiate one such material from another. The temperature difference between the active and inert materials usually becomes noticeable somewhat below the accepted equilibrium temperature.

The area enclosed by any exotherm or endotherm is a function of the weight of reacting material in a sample. The larger the amount of reactant, the larger the area. In other words, the total heat effect is equal to the product of the specific heat of reaction and the weight of the reacting substance. The heating rate does not control the area but it does influence the shape of the area enclosed by the thermal curve. A lower rate causes a broadening at the base and a reduction in amplitude, whereas a higher rate produces a sharper peak of narrow base and increased amplitude.

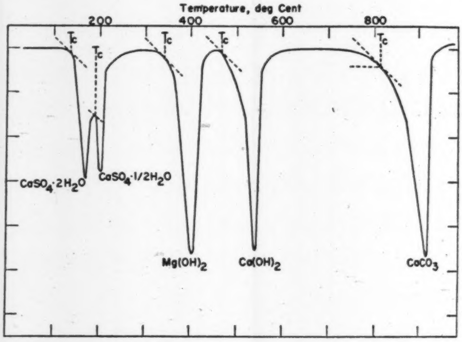

FIG. 1.—White Coat Plaster, Characteristic Temperatures, T_c.

Several methods have been proposed for determining the characteristic temperature of a reaction, but each has its disadvantages as well as advantages. The method for determining the characteristic temperatures, in this study, had previously been found to give satisfactory reproducibility for such compounds as gypsum and calcium and magnesium hydroxides, as well as for carbonates of calcium and magnesium.

This method consists in drawing a tangent, at a 45-deg angle, to the "knee" of an endothermic curve. The temperature at the point of tangency is taken as the characteristic temperature of the material undergoing decomposition. Figure 1 illustrates this method. With the particular scale used in this work (1 in. = 100 C, and 1 in. = 0.2 mv = 5 C) this slope corresponds to this condition where the inert material is heating at a rate of 0.5 C per min faster than the active material.

TABLE I.—CHARACTERISTIC TEMPERATURES OF LIMES USED IN WHITE COAT.

	Hydrate	T_c, deg Cent	
		$Ca(OH)_2$	$Mg(OH)_2$
Dolomitic Hydrated Limes (Type S)	A-1	495	345
	B-1	480	345
	C-1	490	340
	D-1	480	335
	E-1	490	345
	F-1	505	340
Dolomitic Hydrated Limes (Type N)	A-2	475	345
	B-2	485	355
	C-2	480	...[a]
	D-2	480	335
	E-2	485	340
	F-2	500	340
High-Calcium Hydrated Limes	M	495	...[a]
	N	495	...[a]
	O	495	...[a]
	P	485	...[a]

[a] Amount too small to determine T_c.

TABLE II.—CHEMICAL ANALYSES OF HYDRATED LIMES.[a]

	Dolomitic Pressure (Type S)						Dolomitic Normal (Type N)						High Calcium			
Compound....	A-1	B-1	C-1	D-1	E-1	F-1	A-2	B-2	C-2	D-2	E-2	F-2	M	N	O	P
ANALYSIS, PER CENT																
CaO	43.1	42.1	43.4	42.1	40.6	40.4	50.2	47.7	49.5	43.7	44.9	48.5	72.4	73.0	72.5	69.6
MgO..............	29.7	30.2	28.8	29.7	29.0	30.3	30.8	33.5	32.4	33.3	33.0	34.1	0.6	0.1	1.3	1.2
Ignition loss......	26.5	26.6	26.9	26.5	27.2	26.8	18.2	17.8	17.3	21.1	19.7	16.4	25.6	24.5	24.3	28.2
CO_2..............	1.0	2.3	2.2	3.3	4.8	1.0	1.5	2.4	2.8	3.0	4.8	1.4	3.4	2.3	2.1	8.4
H_2O (combined)[b]	25.5	22.2	24.7	23.2	22.4	25.8	16.7	15.4	14.5	18.1	14.9	15.5	22.2	22.2	22.2	19.8
SiO_2..............	0.1	0.3	0.25	0.8	0.8	1.4	0.2	0.3	0.25	0.8	1.2	0.84	0.7	1.3	0.8	0.65
R_2O_3..............	0.3	0.6	0.25	0.7	2.0	0.7	0.5	0.3	0.25	0.7	0.9	0.67	0.4	0.6	0.3	0.65
Fe_2O_3..............	0.060	0.071	0.16	0.35	0.29	0.26	0.070	0.073	0.16	0.32	0.32	0.41	0.11	0.086	0.13	0.47
Total..............	99.7	99.8	99.6	99.8	99.6	99.6	99.9	99.6	99.7	99.6	99.7	100.5	99.7	99.5	99.2	100.3
CALCULATED COMPOSITION, PER CENT																
$CaCO_3$............	2.3	5.2	5.0	7.5	10.9	2.3	3.4	5.5	6.4	6.8	10.9	3.2	7.7	5.2	4.8	19.1
$Ca(OH)$	55.2	51.8	53.6	50.1	45.6	51.6	63.8	58.9	59.6	52.7	51.2	61.7	90.0	91.3	91.3	77.8
$Mg(OH)_2$..........	39.2	37.9	37.9	35.6	36.7	43.1	3.9	3.6	0.0	17.2	8.1	1.6	0.9	0.0	0.0	1.7
MgO..............	2.6	4.0	2.6	5.1	3.6	0.5	28.1	31.0	28.8	21.4	27.4	32.5	0.0	0.1	1.3	0.0
CaO.	0.0	0 0	0.0	0.0	0.0	0.0	0.0	0.0	4.4	0.0	0.0	0.0	0.0	1.0	0.7	0.0

[a] Samples were dried at 110 C before analysis. Analyzed and calculated according to ASTM Standard Methods of Chemical Analysis of Limestone, Quicklime, and Hydrated Lime, (C 25–47), 1949 Book of ASTM Standards, Part 3, p. 187.

[b] By difference.

Table I gives the characteristic temperatures of the limes used in this work.

Materials:

In order to make a fairly comprehensive study of white coat plaster, it was felt that more than just a few commercial brands of hydrated lime should be used. Sixteen samples of hydrated lime were obtained from various lime producers in the United States. Six producers of dolomitic hydrates furnished two samples each, one, type S,[3] and one, type N.[3] Three pairs of dolomitic hydrates were from the Ohio Finishing Lime District, two were from Pennsylvania, and one was from New England. In addition, four high-calcium hydrates were also included in these studies.

In one series of tests, powdered calcite was used as a replacement for lime. The purpose of this series was to provide an inert reference material which would not react with the CO_2 of the atmosphere or with the calcium sulfate.

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[3] Standard Specifications for Hydrated Lime for Masonry Purposes (C 207 – 49), 1949 Book of ASTM Standards, Part 3, p. 163.

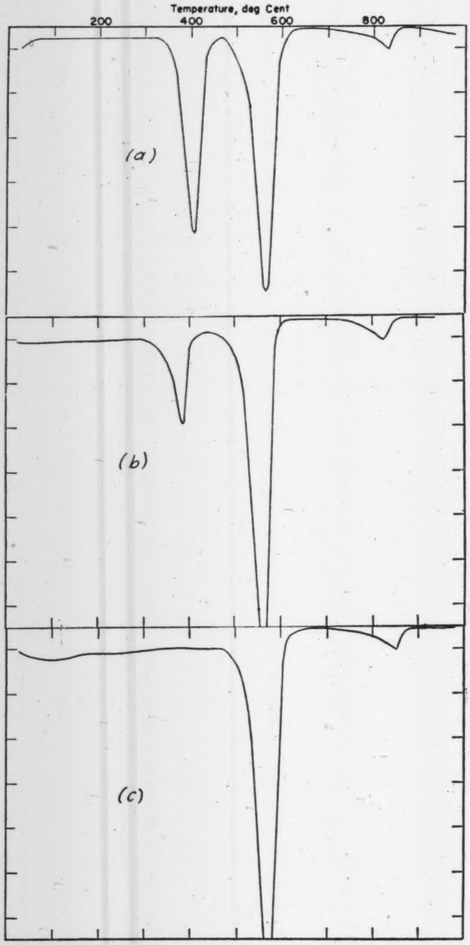

FIG. 2.—Thermograms of the Three General Types of Lime.

The following designators are used to identify the various lime hydrates and the plasters made from these:

A-1, B-1, C-1, D-1, E-1, F-1	dolomitic hydrated limes (type S)
A-2, B-2, C-2, D-2, E-2, F-2	dolomitic hydrated limes (type N)
M, N, O, P	high-calcium hydrated limes
Q	pure calcite

Hydrates A-1 and A-2 were from one manufacturer, hydrates B-1 and B-2 from another manufacturer, etc.

The analysis of these hydrates and their calculated compositions are given in Table II.

Thermograms of the three general types of lime used in this investigation are shown in Fig. 2.

Dolomitic Hydrate (*type S*)—*Fig. 2* (*a*):

The three endotherms represent, from left to right,

$Mg(OH)_2 = MgO + H_2O$ (greater than 92 per cent of MgO hydrated)

$Ca(OH)_2 = CaO + H_2O$

$CaCO_3 = CaO + CO_2$

Dolomitic Hydrate (*type N*)—*Fig. 2* (*b*):

The three endotherms represent, from left to right,

$Mg(OH)_2 = MgO + H_2O$ (very small percentage of MgO hydrated)

$Ca(OH)_2 = CaO + H_2O$

$CaCO_3 = CaO + CO_2$

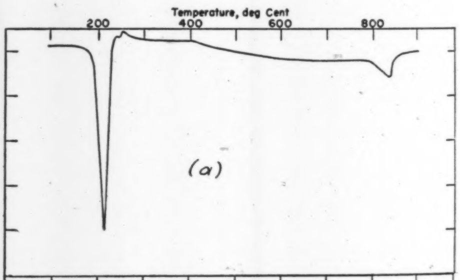

FIG. 3.—Thermograms of Gauging Plaster.

High Calcium Hydrate—Fig. 2 (c):

The two endotherms represent, from left to right,

$Ca(OH)_2 = CaO + H_2O$
$CaCO_3 = CaO + CO_2$

The $CaCO_3$ present in each case is probably due to atmospheric carbonation of a small portion of the $Ca(OH)_2$.

Figure 3 shows thermograms of the gauging plaster as received and after being mixed with water and allowed to set for seven days in air.

Gauging Plaster—Fig. 3 (a):

The first endotherm ($T_c = 180$ C) represents the dehydration:

$$CaSO_4 \cdot \tfrac{1}{2}H_2O = CaSO_4 \text{ (soluble anhydrite)} + \tfrac{1}{2}H_2O$$

The second endotherm, between 800 and 900 C, represents the decomposition of a small amount of limestone ($CaCO_3$) probably present in the original gypsum rock from which the gauging plaster was produced:

$$CaCO_3 = CaO + CO_2$$

The exotherm at about 400 C is caused by the conversion of soluble anhydrite to the insoluble anhydrite form.

Gauged Hemihydrate—Fig. 3 (b):

The first endotherm ($T_c = 90$ C) represents an overlapping of two reactions:

(1) the removal or volatilization of free and adsorbed water

(2) the partial dehydration of dihydrate:

$$CaSO_4 \cdot 2H_2O = CaSO_4 \cdot \tfrac{1}{2}H_2O + 1\tfrac{1}{2}H_2O$$

If (1) had not been present, T_c for (2) would be approximately 140 C.

The second endotherm ($T_c = 195$ C) is due to the dehydration of the hemihydrate which was formed by the thermal dehydration of the dihydrate:

$$CaSO_4 \cdot \tfrac{1}{2}H_2O = CaSO_4 \text{ (soluble anhydrite)} + \tfrac{1}{2}H_2O$$

The third endotherm (800 to 900 C) is similar to that found in the gauging plaster and has been explained.

The exotherm between 300 and 475 C is attributed to the transformation:

$$CaSO_4 \text{ (soluble anhydrite)} = CaSO_4 \text{ (insoluble anhydrite)}$$

Preparation, Storage and Sampling of Plasters:

All of the white coat plasters were prepared by mixing hydrated lime and plaster of Paris in the proportion (by weight) of 70 to 30. The dry mix was then added to a predetermined volume of water to give the proper consistency for application, then thoroughly mixed with a spatula until a smooth, lump-free plaster was obtained. This plaster was applied to 4 by 4 by $\frac{1}{4}$-in. absorptive tile plates by means of a mason's trowel and smoothed to eliminate surface irregularities. The thickness of the white coat was about $\frac{3}{32}$ in.

Prior to the application of the plaster, the plates were soaked in water to prevent the removal of gauging water from the gauged plaster by suction.

Four plates of white coat were prepared from each different mix. One pair of plates was for dry storage, the other pair for wet storage conditions.

One pair of plates from each mix was stored on open racks in a room which was maintained at 70 F. The relative humidity was uncontrolled but averaged approximately 50 per cent. This type of storage condition is designated as dry storage.

The second pair of plates was stored under the same conditions. However, these plates were removed from the racks twice each week and immersed in tap water for two minutes. This type of storage condition is designated as wet storage. It is realized that such a procedure does not simulate conditions on a plastered wall even when the plaster is subjected to unusual dampness caused by excessive condensation of water with temperature and humidity changes. However, this procedure served as an accelerated test, and plasters so treated produced thermograms which were frequently different from those of the same plaster under dry storage conditions.

The analysis schedule was arranged to avoid the necessity of analyzing samples which had insufficient time to lose the excess free water to the atmosphere. No analyses were made on wet storage samples unless at least three days had elapsed between the time of soaking and the analysis.

For differential thermal analysis, approximately 1 sq in. of white coat was removed from each of the two tile plates. The plaster was thoroughly pulverized in a porcelain mortar and then transferred to the active sample well of the analyzer crucible. The well was packed to capacity by repeated filling and tamping, the latter operation being accomplished with the use of a smooth metal plunger.

For the majority of the analyses, the pulverized samples were not weighed. However, since care was taken to grind and pack the samples uniformly in each case, it was felt that variance of sample weight would not be so great as to affect the results obtained in each series except in those cases where a radical change in density of the material had occurred.

Differential thermal analyses were made of the white coat plasters at the following ages after application to the tile plates: 1 day, 7 days, 28 days, 3 months, 6 months, and 12 months.

RESULTS

The results of this work were obtained as a series of thermograms derived from the periodic differential thermal analysis of 17 white coat plasters. These periodic analyses plus the analyses of various related materials totaled approximately 250. Space limitations prevent the presenatation of all the thermograms, but some typical ones are presented to show the application of this method to the study of plaster behavior. Others are presented to illustrate some of the peculiarities which have been found. The conclusions are based upon the studies which have been made of the various combinations taken from the entire group of thermograms.

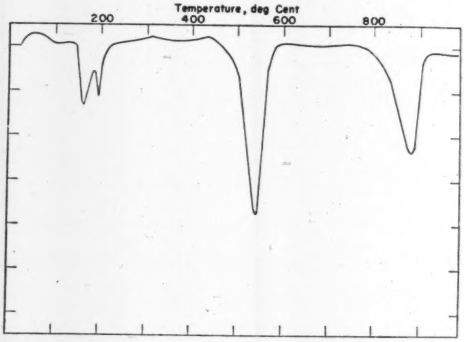

FIG. 4.—White Coat Plaster—One Day Dry Storage.

Normal Plaster:

Figure 4 is the thermogram of a white coat plaster produced from high-calcium hydrated lime. The shallow endotherm in the vicinity of 100 C shows that a small amount of free and adsorbed water is present. The next two endotherms represent the step-wise dehydration of the dihydrate. The large endotherm ($T_c = 475$ C) is due to the $Ca(OH)_2$ whereas the next endotherm ($T_c = 810$ C) represents $CaCO_3$ decomposition. This $CaCO_3$ originates not only from the small amount present in the hydrated lime and in the gauging plaster but also from the atmospheric carbonation of a portion of the $Ca(OH)_2$. (See Figs. 2 and 3).

This may be considered as representative of a normal plaster. There are no indications of other compounds being present. The curves are smooth and regular, without peculiar inflections or doublets, and the size relationships of the endotherms representing the decomposition of the hydrates of calcium sulfate are correct. Many of the thermograms were of this normal type (with the addition of an endotherm representing the decomposition of $Mg(OH)_2$) but others showed irregularities and abnormalities. These irregularities will be considered in detail as they may be indicative of fundamental differences in the hydrates.

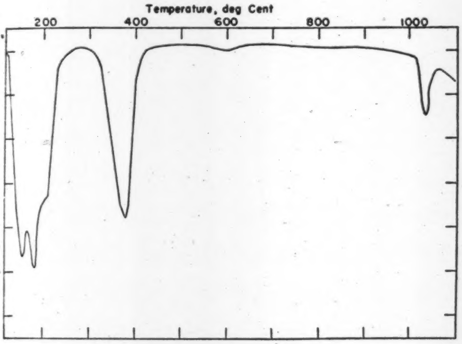

FIG. 5.—Magnesium Sulfate Heptahydrate, $MgSO_4 \cdot 7H_2O$.

Results Under Wet Storage Conditions:

The so-called "wet" storage conditions were selected as constituting a rather mild accelerated test procedure. As the testing progressed, it was evident that unusual reactions were occurring in the plasters and that both gypsum and magnesium hydroxide were being lost.

Bishop (8) reported that hydrated magnesium sulfate efflorescence was formed on white coat plaster containing dolomitic hydrated lime and gypsum when subjected to moist CO_2 and then allowed to dry. The equation for this reaction as given by Bishop is

$$CaSO_4 \cdot 2H_2O + MgCO_3 \cdot 3H_2O + 2H_2O = CaCO_3 + MgSO_4 \cdot 7H_2O$$

However, it has been reported (5, 7) that the formation of $MgCO_3 \cdot 3H_2O$, in the presence of $Ca(OH)_2$, can occur, but only under special conditions. Such conditions may have been met in the case of those white coat plasters subjected to wet storage conditions in this investigation.

Whatever the mechanism may be by which $MgSO_4 \cdot 7H_2O$ is formed, the magnesium hydroxide fraction of a white coat plaster prepared from dolomitic lime must be depleted to some extent, and such a depletion would manifest itself in the production of an $Mg(OH)_2$ endotherm of reduced size. A loss of $CaSO_4 \cdot 2H_2O$ would, of course, also occur.

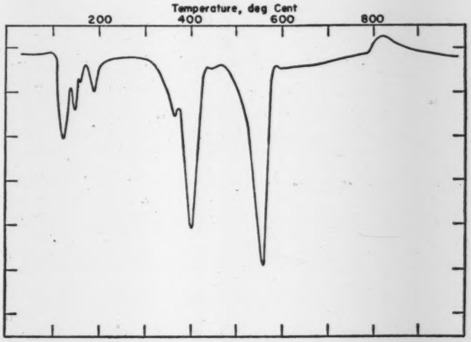

FIG. 6.—$MgSO_4 \cdot 7H_2O$, $CaSO_4 \cdot 2H_2O$, and F-1 Hydrate (1:2:2).

A study of the dolomitic plasters subjected to dry storage conditions did not reveal any loss of $CaSO_4 \cdot 2H_2O$ or $Mg(OH)_2$, but practically every dolomitic plaster subjected to wet storage conditions showed a simultaneous loss of $CaSO_4 \cdot 2H_2O$ and $Mg(OH)_2$. This loss usually became evident at three or six months. Also, in most of these cases the $CaCO_3$ appeared to show an increase, which would be expected if the above reaction occurred.

The opinion is hereby expressed that $MgSO_4 \cdot 7H_2O$ can occur on a wall if sufficient moisture is present. However, the conversion of $Mg(OH)_2$ to $MgSO_4 \cdot 7H_2O$ would occur only in those localized areas in which all of the $Ca(OH)_2$ had first been carbonated.

Figure 5 is the thermogram produced by a sample of $MgSO_4 \cdot 7H_2O$. Figure 6 is the thermogram produced by a dry mixture of $MgSO_4 \cdot 7H_2O$, $CaSO_4 \cdot 2H_2O$, and dolomitic pressure hydrated lime. These thermograms illustrate the overlapping of the endothermic reactions which prevent a positive identification of the magnesium sulfate in dolomitic white coat plaster.

The carbonation of the $Ca(OH)_2$ fraction proceeds with rapidity under wet storage conditions. As a criterion for estimating the rate of carbonation, the amplitude of the $Ca(OH)_2$ endotherm is compared at later ages with that obtained at one day. When the amplitude is less than 10 per cent of the one day amplitude, carbonation is considered to be substantially complete. It should be noted that even at the age of one day, many of the samples appeared to have carbonated up to 50 per cent of the $Ca(OH)_2$ content.

Using this criterion for completeness of carbonation, it was found that 10 of the 16 hydrates showed the $Ca(OH)_2$ portion carbonated at the age of seven days. Hydrates A-1, C-1, D-1, F-1, and A-2 showed complete carbonation at 28 days while hydrate 0 required three months to attain substantially complete carbonation.

Results Under Dry Storage Conditions:

1. *Calcium Sulfate Dihydrate and Hemihydrate.*—It was found that the characteristic temperature, T_c, for the dihydrate is approximately 140 C whereas that of the hemihydrate is approximately 190 C. In several instances, however, the T_c of the dihydrate appeared to be lowered by as much as 15 to 50 C. This apparent lowering is believed to be due to the presence of free or adsorbed water which, upon volatilization during thermal analysis, produces an endotherm which overlaps and to some extent alters the endotherm of the dihydrate.

The thermograms of many of the one and seven day samples showed that there are three separate and distinct endotherms in the temperature range 80 to 200 C. The first peak is undoubtedly due to the above-mentioned free or adsorbed water, the second due to the decomposition of the dihydrate,

$$CaSO_4 \cdot 2H_2O = CaSO_4 \cdot \tfrac{1}{2}H_2O + 1\tfrac{1}{2}H_2O$$

and the third,

$$CaSO_4 \cdot \tfrac{1}{2}H_2O = CaSO_4 \text{ (soluble anhydrite)} + \tfrac{1}{2}H_2O$$

This phenomenon of three separate endotherms was found in the following plasters:

Hydrate	Age
C-1	1 day
D-1	1 day
E-1	1 day
C-2	1 and 28 days
D-2	1 and 7 days
E-2	1 day
F-2	1 and 7 days
N	1 day
O	1 and 7 days

The presence of a large amount of free or adsorbed water at the age of one day is not surprising, but the fact that it only appeared on nine samples and did not appear on eight is of interest. Furthermore, the fact that the adsorbed water persisted in only 4 of 17 samples stored under dry conditions for one week or longer may be significant.

Southard (6) in making dissociation-equilibrium measurements on the system $CaSO_4$-H_2O points out that the approach to equilibrium is a slow process, and considerable time may be required to attain equilibrium conditions. In view of this and the fact that the method of differential thermal analysis produces a curve that is a dynamic and not a static or equilibrium record of a reaction, the peculiarity of three separate endotherms or two with an overlapping reaction in the first becomes more understandable. It is believed that the ability of some white coat plasters to hold larger amounts of water more tenaciously than others is a factor that has a bearing on this matter.

Although the proportions of lime hydrate and gauging plaster were constant in each white coat prepared, five thermograms (C-1, E-1, and E-2 at one day; E-1 at 7 and 28 days) showed an abnormally large endotherm for the reaction,

$$CaSO_4 \cdot 2H_2O = CaSO_4 \cdot \tfrac{1}{2}H_2O + 1\tfrac{1}{2}H_2O$$

However, the endotherm for the reaction,

$$CaSO_4 \cdot \tfrac{1}{2}H_2O = CaSO_4 \text{ (soluble anhydrite)} + \tfrac{1}{2}H_2O,$$

was of normal size which indicates that the first endotherm is not a result of a larger than normal amount of the dihydrate undergoing decomposition since a proportional size-relationship must exist between the di- and hemihydrate endotherms by virtue of the thermodynamic properties of the two substances. Here again, the ability of some plasters to hold free or adsorbed water to a much greater extent than others may be the important factor.

Under normal conditions the endotherm produced by the dehydration of $CaSO_4 \cdot 2H_2O$ is larger in both area and amplitude than that produced by the dehydration of the $CaSO_4 \cdot \frac{1}{2}H_2O$. However, in ten cases (B-1, F-1, D-2, N and O at one day, O at 7 and 28 days, M at 6 months, C-2 and E-2 at 1 yr) the amplitude of the latter is equal to or exceeds that of the former. In those cases where this abnormality occurs in a thermogram produced one day after preparation of the white coat, it is probably due to incomplete hydration of the gauging plaster. This effect is particularly marked in two of the white coats prepared from high-calcium hydrate. A similar effect can be produced by storing over a desiccant. A portion of one white coat was stored under normal atmospheric conditions while another portion of the same material was stored over magnesium perchlorate, a powerful drying agent, in a closed desiccator. Figures 7(*a*) and 7 (*b*) show the thermograms produced by these samples. In the case of the sample stored under normal conditions (Fig. 7 (*a*)), the dihydrate endotherm is larger than that of the hemihydrate, but in the case of the sample stored over the magnesium perchlorate (Fig. 7 (*b*)) the reverse of this situation occurs. The perchlorate has undoubtedly partially dehydrated some of the dihydrate, causing a preponderance of hemihydrate in the sample.

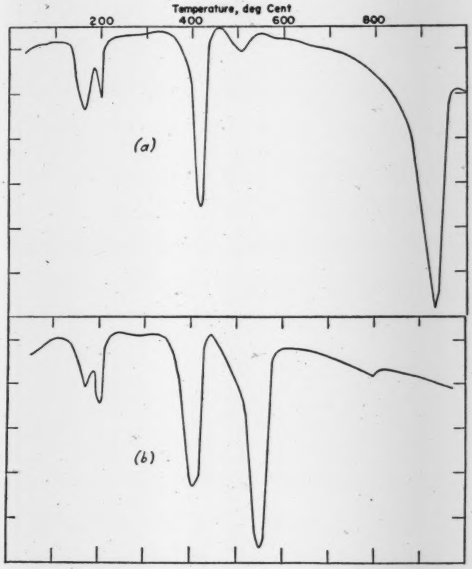

FIG. 7.—White Coat (F-1).
(*a*) 74 days dry storage.
(*b*) 73 days storage over $Mg(ClO_4)_2$.

In three cases (C-2, E-2, and M) the normal size-relationship was found at 7 and 28 days, and then, at 6 months or 1 yr, the hemihydrate endotherm was larger than the dihydrate endotherm. This is believed to be due to atmospheric or internal (within the plaster) dehydration of the dihydrate with the resultant formation of hemihydrate. Such a condition causes the hemihydrate endotherm to increase in relative size because less dihydrate and more hemihydrate is actually present in the white coat plaster than under normal conditions.

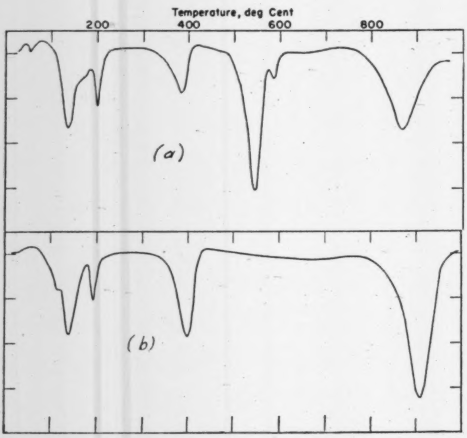

FIG. 8.—White Coat (F-2).
(a) 1 day storage. (b) 7 days storage.

This dehydration effect cannot be attributed to a difference in storage conditions since all 17 plasters were stored in exactly the same manner. The cause of this dehydration is unexplainable at the present time.

A recent publication (10) by Wells and his associates indicated irregularities in the gypsum decomposition range similar to those reported above. Unfortunately, the age of their samples is not known accurately, but presumably most of them were more than 5 years old.

2. *Hydration of Magnesium Oxide.*—Of the six white coat plasters prepared from dolomitic hydrated lime (type N), three showed hydration of MgO to a marked extent. One of these showed a substantial increase in $Mg(OH)_2$ at one day but little, if any, change thereafter. Since this is the period of greatest water content, it is to be expected that if hydration occurs it will do so at this time. The particular lime hydrate used in this plaster (D-2) contained 17.2 per cent $Mg(OH)_2$ and 21.4 per cent MgO according to chemical analysis (see Table II). The percentage of hydrated magnesia in this lime is considerably greater than that of any of the other five Type N limes. Apparently this hydrate was produced from an active quicklime, and the magnesia content is comparatively less difficult to hydrate.

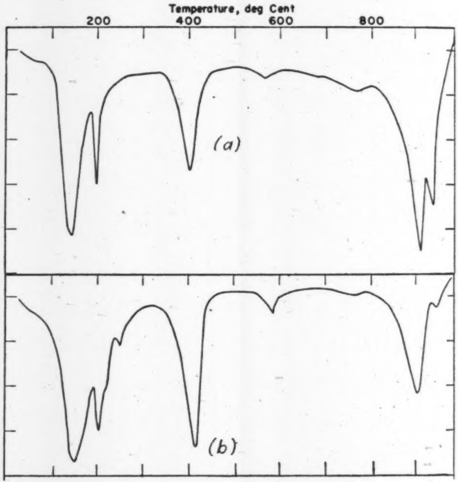

FIG. 9.—White Coat (E-2).
(a) 7 days dry storage. (b) 28 days dry storage.

White coat F-2 (Figs. 8 (*a*) and 8 (*b*)) appears to show some additional hydration of MgO up to one year. The original hydrate contained 1.6 per cent $Mg(OH)_2$ and 32.5 per cent MgO.

White coat E-2 (Figs. 9 (*a*) and 9 (*b*)) gives indication of increasing magnesia hydration up to 1 yr. The lime hydrate in this case analyzed 8.1 per cent $Mg(OH)_2$ and 27.4 per cent MgO.

3. *Calcium Hydroxide.*—Four dolomitic white coat plasters (E-1, C-2, E-2, F-2) when subjected to differential thermal analysis produced a doublet or what appears to be two endotherms in the $Ca(OH)_2$ decomposition range at the age of one day. The T_c in each case was between 565 and 580 C. Figure 8 (*a*) shows this type of doublet. This thermal effect could possibly be an exothermic reaction superimposed on the endothermic decomposition of $Ca(OH)_2$. Such an exotherm would of necessity be of great magnitude and would appear to be improbable. This doublet is probably just what it appears to be at first glance, that is, two separate endothermic reactions which overlap to some extent.

In addition to the above-mentioned doublets, indication of a superimposed reaction was given in two cases by a sudden change of slope or inflection on the $Ca(OH)_2$ endotherm. The hydrates which showed this inflection were A-1 at six months and B-2 at seven days.

4. *Carbonation of Calcium Hydroxide.*—A study of the thermal curves shows that the rate of carbonation of the $Ca(OH)_2$ fraction of the white coat plasters varies with the particular lime hydrate used. The rapidity with which the major portion of the $Ca(OH)_2$ was depleted by $CaCO_3$ formation was quite unexpected, particularly in the case of the one-day tests.

Using the same criterion for rate of carbonation as before, it was found that eight hydrates (C-1, D-1, E-1, D-2, E-2, F-2, M and P) had the $Ca(OH)_2$ content substantially completely carbonated at seven days. Hydrates B-1, F-1, B-2, and O required three months; hydrates A-1 and N required six months; and hydrate C-2 required one year to reach this same degree of carbonation. Hydrate A-2, even after one year, still showed the presence of a considerable amount of $Ca(OH)_2$. There appears to be no relation between the rates of carbonation under dry and under wet conditions.

It should be noted that 15 of the 16 hydrates studied in this investigation were substantially completely carbonated at the end of one year, whereas Wells in his recent report (10) found that most of the 88 plasters analyzed by him still contained appreciable amounts of $Ca(OH)_2$ at much greater ages. This variation in results emphasizes the probable difference in behavior of limes from different manufacturers as being an important factor in determining the behavior on the wall.

5. *Carbonation of Magnesium Hydroxide.*—The carbonation of any $Mg(OH)_2$ in the presence of $Ca(OH)_2$ cannot occur except under very special conditions (5, 7).

The reaction for this carbonation in the presence of $Ca(OH)_2$, if possible under normal conditions, might be:

$$Mg(OH)_2 + CO_2 + 2H_2O = MgCO_3 \cdot 3H_2O$$

$$MgCO_3 \cdot 3H_2O + Ca(OH)_2 = Mg(OH)_2 + CaCO_3 + 3H_2O$$

Thus it can be seen that if any $MgCO_3 \cdot 3H_2O$ could form, the presence of the $Ca(OH)_2$ would cause it to convert to its hydroxide. According to Bishop (8) all of the $Ca(OH)_2$ must first be converted to $CaCO_3$ before any formation of magnesium carbonate can occur. However, Clark *et al* (9) state that $MgCO_3 \cdot 3H_2O$ is stable in the presence of CaO and other calcium compounds.

If it could be assumed that one form or another of magnesium carbonate could be present in the white coat, even if only in localized spots devoid of $Ca(OH)_2$, the thermal effects produced by such would be quite complex and probably be superimposed to such an extent on the normal endotherms produced by $Mg(OH)_2$ and $Ca(OH)_2$ that differentiation could not be made. Figures 10 and 11 illustrate the complexity

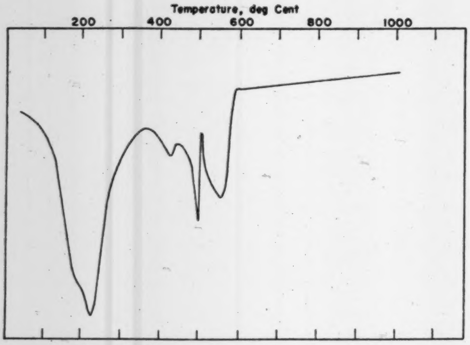

FIG. 10.—Pure Synthetically Prepared Nesquehonite, $MgCO_3 \cdot 3H_2O$.

of the thermal changes which occur when pure, synthetically prepared nesquehonite and natural hydromagnesite are subjected to differential thermal analysis.

Thirteen thermograms produced by the various white coat plasters at different ages showed definite endothermic deviations from the base line in the temperature ranges 225 to 275 C and 600 to 800 C. The hydrates which produced this type of endotherm are:

Hydrate	Age	Approximate Temperature of Reaction	
E-1	28 days	230	740
E-1	3 months	240	...
E-1	6 months	250	...
E-1	12 months	250	710 and 790
E-2	7 days	...	765
E-2	28 days	245	765
E-2	3 months	255	795
E-2	6 months	245	..
E-2	12 months	250	755
F-2	28 days	260	...
F-2	12 months	260	...
O	6 months	260	...
O	12 months	250	...

These endotherms represent reactions which may be, to some extent, characteristic of the thermal decomposition of one or more forms of magnesium carbonate. If these small endotherms are a result of a magnesium carbonate, then the major portion of the thermal curve which normally results from such is undoubtedly masked primarily by the $Mg(OH)_2$ and $Ca(OH)_2$ endotherms.

It has been mentioned that special conditions are necessary to bring about the carbonation of MgO and $Mg(OH)_2$ in the presence of $Ca(OH)_2$. In the case of those white coat plasters stored under wet storage, such special conditions may have been met. However, those plasters stored under dry storage conditions could not meet these special conditions, and yet similar thermal effects were found in both cases. It is probable that carbonation of $Mg(OH)_2$ occurred in the dry storage samples in localized areas in which $Ca(OH)_2$ was absent by virtue of its previous conversion to $CaCO_3$.

6. *Calcium Carbonate.*—All of the hydrated limes used in preparing the white coat plasters contained a small amount of $CaCO_3$ which was due to atmospheric carbonation of some $Ca(OH)_2$.

In six cases (E-1 and D-2 at one day, E-2 at one, seven, 28 days and three months) doublets and inflection points were found in the $CaCO_3$ decomposition range. Figure 9 illustrates this doublet. The reason for the presence of these abnormalities is, as in the case of those in the $Ca(OH)_2$ range, not yet elucidated.

These inflections along with those mentioned in a previous section are undoubtedly due to an overlapping of two reactions and show that differentiation of simultaneously occurring reac-

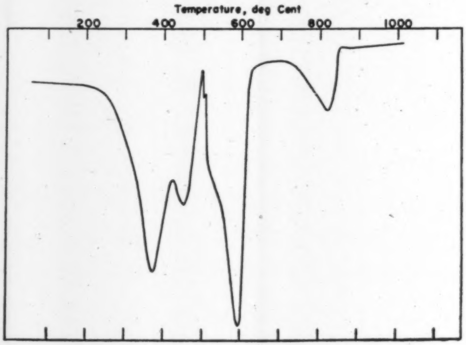

FIG. 11.—Natural Hydromagnesite, $5MgO \cdot 4CO_2 \cdot 5H_2O$.

tions or of those occurring in approximately the same temperature range is impossible by differential thermal analysis. This is perhaps the most serious drawback to this method of analysis.

Calcite-Gypsum Plaster:

In the thermograms produced by analysis of this particular white coat, the calcium sulfate dihydrate and hemihydrate endotherms showed the normal size-relationship throughout the twelve month period and do not exhibit any of the previously discussed abnormalities. However, the T_c for the $CaSO_4 \cdot 2H_2O$ is in all cases approximately 15 to 20 C lower than is found in the majority of the analyses of the other white coat plasters. Such a deviation suggests the possibility that the $CaSO_4 \cdot 2H_2O$ gel structure is more stable and retains its adsorbed water for much longer periods of time when lime is not present. The basis for this reasoning lies in the fact that cations, particularly multivalent cations, such as Ca^{++} and Mg^{++}, accelerate the set of gypsum and that an excessive amount of cations, above the coagulation value, will coagulate the gel, bring about crystallization, and reduce the residual forces which hold the excess water. The cation effect is diminished greatly in the case of calcite with the absence of lime and magnesia since the number of cations resulting from the difficulty soluble $CaCO_3$ is of a much lower order of magnitude than that in the presence of the more soluble $Ca(OH)_2$.

$\bar{X}$, the average T_c for the $CaCO_3$ in the calcite-gypsum plasters subjected to dry and wet storage conditions was 845 C with a standard deviation, σ, of 8.2 C. The $\bar{X}$ for the $CaCO_3$ in the lime-gypsum plasters varied with age from 807 to 832 C. The reason for the higher T_c for the calcite lies in its larger particle size and its very high degree of purity. The carbonate in the lime-gypsum plaster is primarily a precipitated product and as such is of a finer state of subdivision.

Differential thermal analysis of the pure calcite which was used in this special plaster gave a T_c of 895 C. As can be seen this is higher than for the calcite in the calcite-gypsum plaster which, in turn, is higher than for the carbonate in the lime-gypsum plasters. The variation in T_c is undoubtedly due to a variation of particle size.

It is of particular interest to note that although there was a continuous loss of $CaSO_4 \cdot 2H_2O$ with time for all lime-gypsum plasters stored under wet conditions, no such loss occurred in the calcite-gypsum plaster. It has been previously hypothesized that the gypsum gel structure is more stable in the absence of lime or, more specifically, in the absence of calcium ions. As a consequence of this stability of gel structure, the solubility of the $CaSO_4 \cdot 2H_2O$ is believed to be reduced and hence it is not leached out of the calcite-gypsum plaster under wet storage conditions.

SUMMARY

Plasters Containing Dolomitic Hydrates—Type S:

The six brands of this type of dolomitic hydrate were not very dissimilar chemically, yet they showed relatively large differences in the rate of carbonation of the calcium hydroxide fraction.

Hydrate	Time for Substantially Complete Carbonation
A-1	6 months
B-1	3 months
C-1	7 days
D-1	7 days
E-1	7 days
F-1	3 months

Apart from the variation in the rate of carbonation, all plasters of this type, except E-1, showed only minor irregularities or variations from the so-called normal plaster. Hydrate E-1 exhibited numerous irregularities at all ages. These irregularities were found in the gypsum decomposition range, in the possible presence of a carbonate of magnesium, and in the behavior of both $Ca(OH)_2$ and $CaCO_3$ during decomposition.

Plasters Containing Dolomitic Hydrates—Type N:

The six brands of this type of dolomitic hydrate differed appreciably in their calculated chemical composition, particularly in regard to the degree of hydration. In this property the range was from 4.4 per cent unhydrated CaO to 17.2 per cent $Mg(OH)_2$. The rates of carbonation of the $Ca(OH)_2$ fraction varied even more than that found for the type S hydrates as is shown in the following tabulation:

Hydrate	Time for Substantially Complete Carbonation
A-2	>1 year
B-2	3 months
C-2	1 year
D-2	7 days
E-2	7 days
F-2	7 days

In only one case (hydrate F-2) was the time for carbonation of the type N hydrate shorter than was found for the type S hydrate from the same manufacturer (F-1).

Three of these hydrates (A-2, B-2, and C-2) gave no indication of any increase in the amount of hydration of MgO at ages up to one year. Hydrate D-2 showed appreciably more hydration of MgO in the plaster at the age of one day, but no additional hydration was evident after the one day test. Hydrates E-2 and F-2 gave indications of continued hydration of MgO up to and including the one year tests.

Hydrate E-2 showed many deviations from the normal thermograms at all ages. In this respect it was very similar to the type S hydrate (E-1) from the same producer. The other five hydrates showed very few deviations from the normal, and those that were found occurred primarily at the one-day or seven-day test period.

Plasters Containing High Calcium Hydrates:

The carbonation of the $Ca(OH)_2$ in these hydrates varied as is shown below:

Hydrate	Time for Substantially Complete Hydration
M	7 days
N	6 months
O	3 months
P	7 days

Abnormalities in these hydrates, other than those connected with the hydration of gypsum, were few.

Retention of Adsorbed Water:

In 9 of the 16 hydrates, evidence of retention of free or adsorbed water was obtained at the one-day age. This indicates that about half the plasters held water more tenaciously than the others. These nine cases were scattered uniformly over the three types of lime and may have been due to minor variations in the preparation of the plasters. However, three plasters (D-2, F-2, O) still held adsorbed water at the age of 7 days, and one (C-2) still evidenced the presence of adsorbed water at 28 days. It should be noted that all three of these plasters that retained adsorbed water beyond the one-day period were made from type N dolomitic hydrates.

Hydration And Dehydration of Gypsum

Since one lot of gauging plaster was used throughout all of these tests, any variations found in the hydrates of calcium sulfate must be attributed to specific action of the limes used. While most of the tests indicated perfectly normal gypsum characteristics, a few showed that the calcium sulfate had not completely hydrated at one day. This condition was found with hydrates B-1, F-1, D-2, N, and O. In the case of hydrate O, the indications were that normally complete hydration of the $CaSO_4$ had not occurred at 28 days, although it was complete at 3 months.

In a few cases, hydration of $CaSO_4 \cdot \frac{1}{2}H_2O$ was apparently normal at early ages, but at later ages evidence was obtained indicating that the amount of dihydrate present was decreasing and the amount of hemihydrate present was increasing. In a few other cases, indications were found that a similar desiccation was occurring, but the evidence was not conclusive at the one-year period.

CONCLUSIONS

1. Atmospheric carbonation of the calcium hydroxide fraction of white coat plasters occurs with great rapditiy, particularly during the first several days of exposure.

2. The rate of calcium hydroxide carbonation appears to vary considerably with the particular brand and type of lime hydrate in the plaster.

3. Positive evidence pointing to the carbonation of the magnesium hydroxide fraction could not be found because of the complexity of the thermal effects produced by the various forms of magnesium carbonate. However, several unexplainable peculiarities in the thermograms of white coat plaster show some resemblance to certain portions of the thermal curves produced by carbonated magnesia.

4. The retention or persistence of free or adsorbed water in aged white coat plaster varies considerably.

5. In several cases a definite increase in the calcium sulfate hemihydrate was observed after several months. This increase is undoubtedly due to the dehydration of a portion of the dihydrate.

6. The hydration of MgO in plasters prepared from type N hydrates varies considerably with the particular brand of hydrate.

7. As in the case of magnesium carbonate, positive evidence for the presence of magnesium sulfate in the white coat plaster is lacking, but a decided loss of both $CaSO_4 \cdot 2H_2O$ and $Mg(OH_2)$ along with an increase of $CaCO_3$ in those plasters subjected to wet storage conditions leads to the belief that magnesium sulfate does form in white coat plasters although probably only in localized areas depleted of $Ca(OH)_2$.

8. The results of this investigation neither discredit nor substantiate the theory which places the blame for plaster failures solely on the presence of unhydrated magnesia and its subsequent hydration to $Mg(OH)_2$ with disruptive expansion. However, it is felt that several other factors should be considered in connection with plaster failures:

(*a*) The partial dehydration of gypsum on a wall as a function of temperature and humidity.

(*b*) The factors influencing precipitation conditions and the growth of precipitated particles.

(*c*) The calcium compounds, their physical condition, and the chemical reactions in which they take part or are produced.

Acknowledgment:

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REFERENCES

(1) E. S. Newman, "Investigations of Plaster Failure," *Bulletin*, Am. Ceramic Soc., Vol. 26, No. 4, p. 117 (1947).

(2) D. F. Richardson, "Analysis of Hydrated Limes by a Thermo-Chemical Method," *Industrial and Engineering Chemistry*, May, 1927. p. 625.

(3) L. S. Wells and K. Taylor, "Hydration of Magnesia in Dolomitic Limes," *Journal of Research*, Nat. Bureau of Standards, Vol. 19, No. 2 (1937) (*RP*1022).

(4) L. S. Wells, W. F. Clarke, and E. M. Levin, "Expansive Characteristics of Hydrated Limes and the Development of an Autoclave Test for Soundness," *Journal of Research*, Nat. Bureau of Standards, Vol. 41 (1948) (*RP*1917).

(5) G. L. Clark, R. E. Grim, R. S. Sprague, W. W. Sprague, "Hydration of Lime and Its Relation to Plaster Failures," unpublished.

(6) K. K. Kelley, J. C. Southard, and C. T. Anderson, "Thermodynamic Properties of Gypsum and Its Dehydration Products," *Technical Paper 625*, Bureau of Mines (1941).

(7) R. S. Sprague, Doctorate Dissertation, University of Illinois (1949).

(8) D. L. Bishop, "Function of Carbon Dioxide in Producing Efflorescence on Plaster and Cement Products," *Journal of Research*, Nat. Bureau of Standards, Vol. 30, No. 5, p. 361 (1943) (*RP*1538).

(9) G. L. Clark, P. M. Bernays and J. P. Tordella, "Progress in Lime Investigations with X-Rays and the Electron Microscope," *Proceedings*, National Lime Association (1942).

(10) L. S. Wells, W. F. Clarke, Edwin S. Newman and D. L. Bishop, "Investigation of Failures of White-Coat Plaster," Nat. Bureau of Standards, Building Materials and Structures Report 121.

IDENTIFICATION OF CLAY MINERALS BY STAINING TESTS*

By Richard C. Mielenz[1] and Myrle E. King[1]

Synopsis

Staining tests make possible rapid identification of clay minerals in soils and rocks. Two types of reactions are indicated: (1) acid-base reactions in which stains act as indicators of pH in adsorbed water films and (2) oxidation-reduction reactions in which certain aromatic amines are partially oxidized, with production of characteristically colored forms. Best results are obtained with safranine Y and malachite green in nitrobenzene solution for acid-base reactions, and with water solution of benzidine in oxidation-reduction reactions. Samples should be acid-treated and objectionable quantities of iron oxides and organic materials removed prior to the tests.

Acid-base reactions are rapid and reliable for identification of montmorillonite-type minerals (except hectorite), illite, and halloysite in proportions greater than about 5 per cent. Kaolinite and anauxite can be distinguished from nacrite and dickite by induced pleochroism. New tests with benzidine reliably separate illite from montmorillonite-type minerals, palygorskite (attapulgite), the kaolinite minerals, and the halloysites; and anauxite can be distinguished from kaolinite. Consequently, acid-base and oxidation-reduction reactions are complementary staining methods.

Procedures, applications, and limitations of the staining methods are indicated. Data regarding adsorption of dyes by clays and mechanisms of the staining reactions are summarized.

Staining tests are useful in microscopy because they facilitate mineral identification and increase the rapidity and accuracy of mineralogical analysis through development of characteristic and easily perceived colors in individual mineral particles. Organic and inorganic stains have been used to identify many minerals but they find their best application in clay mineral identification because the mineralogic complexity and typical fineness of grain size of clays preclude adequate study by ordinary petrographic methods.

Staining techniques applied to clays depend upon two physico-chemical phenomena (1) oxidation-reduction reactions and (2) acid-base reactions, both types of reaction accompanying adsorption of the staining agent by the clay particles (1).[2] The color transformations effected by the oxidation-reduction reactions usually are markedly influenced by changes in pH value of the applied solution. Several investigators have attempted to develop diagnostic criteria for recognition of clay minerals through measurement or observation of adsorption of dyes, principally malachite green, by soils without regard to oxidation-reduction or acid-base reactions (2, 3, 4, 5). However, without prior knowledge of the proportion of clay, identification of the constituent clay minerals by this procedure is most uncertain. Effect of dye adsorption by clay minerals in suspension upon absorption of transmitted light shows promise in clay mineral identification (6).

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[1] Head, Petrographic Laboratory, and Petrographer, respectively, Bureau of Reclamation, Denver, Colo.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1231.

This paper will examine the staining reactions of clay minerals and describe specific procedures by which generally reliable identification and semiquantitative analysis of clays and soils can be accomplished. In attempting to evaluate the reliability of any method of clay mineral identification, the complex mineralogic relationships existing among the clay minerals should be kept in mind.

Present knowledge is consistent with a classification of the most common clay and claylike minerals as follows:

MICACEOUS CLAY AND CLAYLIKE MINERALS

Ratio of Silica to Aluminous Molecular Layers 1:1 (7)

- *Kaolinite Group*
  - Kaolinite
  - Dickite
  - Nacrite
  - Anauxite
- *Halloysite Group*
  - Halloysite, $2H_2O$ (syn. Metahalloysite)
  - Halloysite, $4H_2O$ (syn. Endellite, Hydrated Halloysite)

Ratio of Silica to Aluminous, Magnesian, or Ferriferous Molecular Sheets 2:1 (7)

- *Montmorillonite Group*
  - Heptaphyllitic (8)
    - Montmorillonite
    - Beidellite
    - Nontronite
  - Octophyllitic (8)
    - Saponite
    - Hectorite
- *Hydrous Mica Group*
  - Hepaphyllitic (8)
    - Illite
    - Pinite
  - Octophyllitic (8)
    - Glauconite
    - Celadonite
    - Vermiculite

AMPHIBOLIC CLAY AND CLAYLIKE MINERALS

- *Palygorskite Group*
  - Palygorskite (attapulgite)

AMORPHOUS CLAY MINERALS

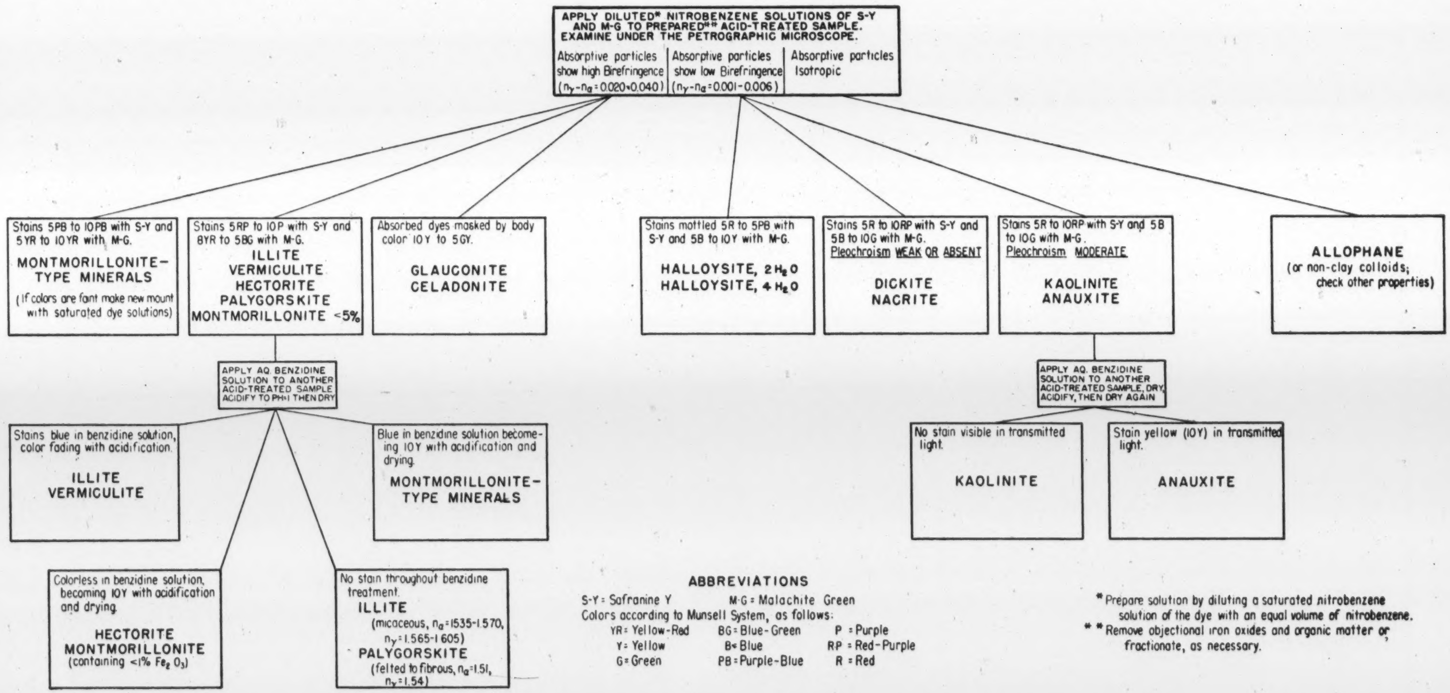
- *Allophane Group*
  - Allophane

General treatments of the clay minerals as a whole have been published by Grim (9) and by Kerr (10).

A consideration of clay minerals should include several minerals which commonly occur with clay minerals, possess properties similar to those of certain established clay minerals, are crystallographically and compositionally similar to clay minerals, and which are separable from clay minerals only by application of arbitrary criteria. Minerals in this category are glauconite, celadonite, vermiculite, and pinite.

A satisfactory staining test should permit identification of at least most individual species of the clay and claylike minerals. However, gradations exist between montmorillonite, nontronite, and beidellite (11); probably between illite and the montmorillonite group; and at least to some extent between kaolinite and halloysite (12). Moreover, all gradations apparently exist between illite and muscovite, and gradations of members of the illite and montmorillonite group to the claylike minerals glauconite, celadonite, and vermiculite may be established subsequently.

In addition to progressive intergradation through changes in chemical composition and crystallinity, interlayering of clays by intercalation of molecular sheets of differing composition in the same clay crystal has been demonstrated. Interlayering of illite and montmorillonite (13, 14), of kaolinite and nontronite (15), and possibly of glauconite and montmorillonite (16) has been identified. More-


APPLY DILUTED* NITROBENZENE SOLUTIONS OF S-Y AND M-G TO PREPARED** ACID-TREATED SAMPLE. EXAMINE UNDER THE PETROGRAPHIC MICROSCOPE.
Absorptive particles show high Birefringence ($n_\gamma - n_\alpha = 0.020 - 0.040$)
Absorptive particles show low Birefringence ($n_\gamma - n_\alpha = 0.001 - 0.006$)
Absorptive particles Isotropic
Stains 5PB to 10PB with S-Y and 5YR to 10YR with M-G.
MONTMORILLONITE-TYPE MINERALS
(If colors are faint make new mount with saturated dye solutions)
Stains 5RP to 10P with S-Y and 8YR to 5BG with M-G.
ILLITE
VERMICULITE
HECTORITE
PALYGORSKITE
MONTMORILLONITE <5%
Absorbed dyes masked by body color 10Y to 5GY.
GLAUCONITE
CELADONITE
Stains mottled 5R to 5PB with S-Y and 5B to 10Y with M-G.
HALLOYSITE, $2H_2O$
HALLOYSITE, $4H_2O$
Stains 5R to 10RP with S-Y and 5B to 10G with M-G.
Pleochroism WEAK OR ABSENT
DICKITE
NACRITE
Stains 5R to 10RP with S-Y and 5B to 10G with M-G.
Pleochroism MODERATE
KAOLINITE
ANAUXITE
ALLOPHANE
(or non-clay colloids; check other properties)
APPLY AQ. BENZIDINE SOLUTION TO ANOTHER ACID-TREATED SAMPLE ACIDIFY TO PH=1 THEN DRY
Stains blue in benzidine solution, color fading with acidification.
ILLITE
VERMICULITE
Blue in benzidine solution becoming 10Y with acidification and drying.
MONTMORILLONITE-TYPE MINERALS
Colorless in benzidine solution, becoming 10Y with acidification and drying.
HECTORITE
MONTMORILLONITE
(containing <1% Fe_2O_3)
No stain throughout benzidine treatment.
ILLITE
(micaceous, n_α=1535-1.570, n_γ=1.565-1.605)
PALYGORSKITE
(felted to fibrous, n_α=1.51, n_γ=1.54)
APPLY AQ. BENZIDINE SOLUTION TO ANOTHER ACID-TREATED SAMPLE, DRY, ACIDIFY, THEN DRY AGAIN
No stain visible in transmitted light.
KAOLINITE
Stain yellow (10Y) in transmitted light.
ANAUXITE

ABBREVIATIONS

S-Y = Safranine Y   M-G = Malachite Green

Colors according to Munsell System, as follows:

YR = Yellow-Red	BG = Blue-Green	P = Purple
Y = Yellow	B = Blue	RP = Red-Purple
G = Green	PB = Purple-Blue	R = Red

* Prepare solution by diluting a saturated nitrobenzene solution of the dye with an equal volume of nitrobenzene.

** Remove objectional iron oxides and organic matter or fractionate, as necessary.

FIG. 1.—Identification of Clay Minerals by Staining Tests.

over, interlayering of vermiculite, biotite, and chlorite is known (17, 18). Additional combinations of the micaceous clay and claylike minerals, micas, and chlorites probably will be discovered.

Staining tests for mineral identification are supplements to other petrographic techniques. They can be used successfully only if combined with determination of other mineralogic properties, including index of refraction, birefringence, particle shape, cleavage, pleochroism, color, and other characteristics.

To make possible objective reporting of colors developed in the tested materials by the staining reactions, the Munsell system of color notation (19) is used throughout this report. A summary of the system is given in Appendix I.

Procedures of recommended staining tests are presented in Appendices III and IV. The recommended sequence of staining tests is summarized in Fig. 1. Methods of sample preparation are indicated in Appendix II.

EVALUATION OF STAINING TESTS FOR IDENTIFICATION OF CLAY MINERALS

Staining tests to identify clay minerals occurring in rocks and soils can be evaluated by several criteria, as follows:

1. Development in particles of clay of colors which are: (*a*) reproducible, (*b*) sufficiently characteristic to permit identification of one or more clay mineral species, (*c*) sufficiently intense to permit ready perception, (*d*) preferably of hues not common in the minerals themselves.
2. Preliminary treatment of samples not laborious.
3. Interferences uncommon.
4. Conditions favorable to development of characteristic colors easily controlled.

ADSORPTION OF STAINS BY CLAY MINERALS

Staining reactions of clay minerals depend upon (*a*) the adsorptive capacity of the various clay minerals for dye molecules, (*b*) the position occupied in the clay crystal by the adsorbed dye molecule, and (*c*) the adsorption actions brought into play.

Adsorptive Capacity of Clay Minerals for Dyes:

Only limited data on adsorption capacity of clay minerals for dyes are available. In recent work, Bosazza (3) shows that 1 g of each of three kaolins adsorbed 0.0074 to 0.0082 g of malachite green, 0.0073 to 0.0081 g of congo red, and 0.0083 g of methyl violet. In the same test 1 g of Wyoming bentonite adsorbed 0.036, 0.045, and 0.047 g of these dyes. Cohen and Knight (2) demonstrated that very small samples (0.2 g) of soils containing varying proportions of different clay minerals adsorb 0.003 to 0.30 g of malachite green per gram.

In tests in the Petrographic Laboratory, using saturated solutions of safranine Y and malachite green in nitrobenzene, six samples containing several clay minerals were found to adsorb 0.0154 to 0.0167 g of safranine Y and 0.0188 to 0.0925 g of malachite green per gram of clay (Table I). The safranine Y solution was depleted of safranine Y by all the clays during the tests; consequently, all the indicated adsorptions for this dye probably are much less than the capacity of the samples.

Benzidine is the most widely used compound in oxidation-reduction reactions of clays. Adsorption tests by Weil-Malherbe and Weiss (1) indicated that acid-treated Wyoming bentonite adsorbs 0.040 to 0.063 g (0.22 to 0.34 millimole) of benzidine per gram of sample from benzene solutions, in accordance with the Freudlich adsorption isotherm. Acid-treated montmorillonite adsorbed 0.092

to 0.168 g of benzidine per gram of sample from an aqueous solution heated to boiling and shaken for 30 min; the maximum amount of benzidine adsorbed by a sample with a cation exchange capacity of 0.91 me per g was 0.91 millimole per g (20).

Location of Adsorbed Dye Molecules in Clay Crystals:

Adsorbed organic molecules or ions are thought to accumulate on the surface of crystals, with certain crystal faces adsorbing far more than others. Gieseking (21), Hendricks (20), and Bradley (22) have concluded that many kinds of organic molecules are adsorbed along the (001) plane of montmorillonite between successive silicate packets; and they demonstrated a relationship between *d*(001) for the clay crystal and the kind and amount of organic compound adsorbed. In particular, Hendricks (20) and Bradley (22) concluded that benzidine adsorbed by montmorillonite from aqueous

TABLE I.—ADSORPTION OF DYES FROM SATURATED NITROBENZENE SOLUTION.[a]

Sample[a]	Approximate Clay Content	Dye Adsorbed, g per g	
		Safranine Y [b]	Malachite Green
Kaolinite Mesa Alta, New Mexico (API No. 9a)	Kaolinite 90 per cent	0.0154	0.0188
Halloysite Eureka, Utah (API No. 13)	Halloysite 96 per cent,[a] montmorillonite—trace	0.0164	0.0390
Illite Fithian, Ill. (API No. 35)	Illite 75 per cent, kaolinite 1 to 2 per cent.	0.0167	0.0343
Montmorillonite Otay, Calif. (API No. 24)	Montmorillonite 97 per cent	0.0166	0.0650
Montmorillonite Belle Fourche, S. D. (API No. 27)	Montmorillonite 85 per cent	0.0155	0.0925
Nontronite Garfield, Wash. (API No. 33a)	Nontronite 100 per cent	0.0156	0.0502

[a] 0.2 g acid-treated sample passing No. 200 sieve and dried at 105 C for 24 hr immersed in 10 ml saturated solution of dye in nitrobenzene for 1 hr, with occasional agitation, at room temperature. Concentrations of dyes in original solutions were 339 mg per liter safranine Y and 1935 mg per liter malachite green. Samples selected from reference clays, Project 49, American Petroleum Institute (39).

[b] Low magnitude and constancy of adsorption is caused by small amount of dye remaining in solution, ranging from 6.5 to 31.6 mg per liter at completion of the test.

TABLE II.—EFFECT OF STAINING TREATMENTS ON 001 SPACING OF MONTMORILLONITE.

Staining Agent	Staining Treatment	Treatment before X-ray Diffraction Exposure	001 Spacing, Å		
			Ca-Montmorillonite[a]	H-Montmorillonite[a]	Na-Montmorillonite[a]
None	None	Room drying	13.1[b]	12.1[b]	11.0[b]
		4 hr at 105 C	12.1[c]	...	...
		4 hr at 151 C	10.4[c]	...	...
		4 hr at 190 C	10.0[c]	9.8[c]	...
Saturated water solution of benzidine	4 hr in slight excess of solution	Wet with water	19.7[c]	...	...
		Room drying	13.8[b]	...	...
		4 hr at 190 C	10.1[c]	...	...
	4 hr in great excess of solution (about 1 g in 50 ml) at room temperature	5 days room drying	13.2[b]	13.9[b]	11.8 and 15.1[b,d]
		5 days room drying and 4 hr at 190 C	11.0[c]	13.4 to 14.7[c]	9.8[c]
	4 hr in great excess of solution (about 1 g in 50 ml) at about 90 C	6 days room drying	13.9[b]	15.1[b]	11.8 and 15.0[b,d]
		6 days room drying and 4 hr at 190 C	13.8 to 13.9[c]	15.1[c]	9.8 and 15.1[c,d]
Saturated water solution of safranine Y	2 to 5 min in slight excess of solution	Drying at about 50 C	...	13.1[c]	...
		Drying at about 50 C and 4 hr at 190 C	...	9.9[c]	...
		4 hr at 190 C	9.9[c]	10.1[c]	...
Nitrobenzene	2 to 5 min in slight excess of nitrobenzene	None	15.4[c]	15.3[c]	...
		Room drying	15.4[b]	...	...
		4 hr at 190 C	...	9.8[c]	...
Saturated nitrobenzene solution of safranine Y	2 to 5 min in slight excess of solution	None	15.4[c]	15.3[c]	...
		Room drying	15.4[b]	...	...
		4 hr at 90 C	...	9.8[c]	...

[a] Ca-Montmorillonite and Na-Montmorillonite supplied by R. E. Grim; H-Montmorillonite is acid-treated, Na Montmorillonite, Osage, Wyoming (API No. 26 (39)).

[b] Sample extruded from stainless steel capillary for X-ray diffraction exposure.

[c] Sample packed in glass capillary and sealed for X-ray diffraction exposure.

[d] Two diffraction rings clearly observed.

solutions is held in this position with the plane of the biphenyl nucleus parallel to the (001) plane of the montmorillonite. Dried samples of montmorillonite previously tested with benzidine solution showed $d(001)$ of 13.0 A and 15.2 Å, and were considered to contain one and two layers, respectively, of benzidine molecules between adjacent silicate packets.

However, X-ray diffraction analysis of calcium, hydrogen, and sodium montmorillonites indicates that little, if any, benzidine or safranine Y penetrates the (001) interlayer space under usual conditions of the staining tests (Table II). For example, although $d(001)$ of room-dry Ca-montmorillonite, after treatment with a saturated aqueous solution of benzidine in slight excess of saturation, differs from that of the untreated minerals (13.8 A against 13.1 Å), heating at 190 C, a temperature found to remove virtually all reversible adsorbed water (23), indicates that the difference results from differences in the adsorbed water films. On the other hand, during treatment of montmorillonite with a considerable excess of the benzidine solution at a near-boiling temperature, the benzidine molecule is introduced along the 001 interlayer space. Benzidine introduced into H-montmorillonite by melting the solid benzidine at about 150 C also develops two molecular layers in the interlayer space, for $d(001)$ measures 15.7 Å.

These determinations indicate that under conditions prevailing in the staining tests of clay minerals, the adsorbed dye is held almost entirely on the exterior of the clay crystals, probably with the main concentration upon the 001 planes exposed at the ends of the crystal or along partings within the crystal. These partings may be of submicroscopic dimensions. The molecules of dye adsorbed on these surfaces are oriented to produce the pleochroic effects evident in some clays when examined in plane polarized light under the microscope (24, 25).

Mechanism of Adsorption of Dyes by Clay Minerals:

Gieseking (21) and others (20, 22) have demonstrated that certain organic cations participate in cation exchange reactions of clay minerals. Grim, Allaway, and Cuthbert (26) concluded that small-size organic cations exchange with inorganic cations and are not adsorbed in excess of the cation exchange capacity of the clay. On the other hand, although large organic cations participate in exchange reactions of clays, amounts added in excess of the exchange capacity of the clay also are adsorbed, probably under the influence of van der Waals' forces.

Experiments performed in the Petrographic Laboratory indicate that benzidine is adsorbed without exchange of the hydrogen ion in acid-treated montmorillonite, but that adsorption of safranine Y and probably of malachite green leads to displacement of the hydrogen ion in the same manner as does the Na^+ ion from dissolved sodium chloride (Table III). Under the indicated conditions the benzidine apparently combines with the adsorbed H^+ of the clay in a neutralization reaction. Although adsorbed H^+ is neutralized by adsorbed benzidine without exchange, Na^+ of Wyoming bentonite is replaced at least in part by benzidine.

STAINING INVOLVING OXIDATION-REDUCTION REACTIONS

Oxidation of Amines by Clay Minerals:

Oxidation of certain aromatic amines produces a series of compounds of varying stability, some of which are strongly colored. Hendricks and Alexander (27) and Hauser and Leggett (28) showed independently in 1940 that colored amine-clay complexes could be formed following adsorption by montmorillonite clays of the original reduced amine, presumably as the result of partial oxidation of the adsorbed amine by ferric ions bound to the clay crystal or release of atomic oxygen (29). Hauser and Leggett (28) tabulate color reactions obtained between a Wyoming bentonite and 46 different amine compounds. They conclude:

TABLE III.—CHANGE OF pH OF SLURRY OF ACID-TREATED BENTONITE WITH ADDITION OF VARIOUS AQUEOUS SOLUTIONS.[a]

Solution Added, ml	pH of Slurry			
	NaCl Solution[b] (0.5 N Aq.)	Safranine Y[b] (Sat'd. Aq.)	Malachite Green[b] (0.16 per cent Aq.)	Benzidine[b,c] (Sat'd. Aq.)
0.00	4.0	4.2	4.4	4.5
0.05	3.4	4.4	4.2	4.4
0.10	3.3	4.4	4.1	4.4
0.15	3.3	4.3	4.1	4.4
0.20	3.2	4.3	4.1	4.4
0.30	3.2	4.2	4.0	4.4
0.50	3.2	4.1	3.9	4.4
0.75	3.2	4.0	3.8	4.4
1.00	3.2	3.9	3.8	4.5
1.50	3.2	3.8	3.7	4.5
2.00	3.2	3.8	3.6	4.6
3.00	3.2	3.7	3.4	4.7
5.00	3.2	3.6	3.3	4.9
7.50	3.2	...	3.2	5.1
10.00	3.2	3.6	3.1	5.4
15.00	3.2	3.6	3.1	5.8
20.00	...	...	3.0	6.0
25.00	...	3.6	3.1	6.2

[a] 0.200 g room-dry acid-treated Wyoming bentonite (API No. 26 (39)) in 35 ml distilled water; portions of various solutions added slowly, mixed thoroughly, and pH read on Beckman pH meter with slurry in agitated condition.

[b] pH of solutions as follows: NaCl, 6.3; safranine Y, 5.1; malachite green, 3.0; benzidine, 6.7.

[c] Aqueous slurry is milky, with addition of benzidine solution becomes progressively pale yellow, after 0.2-ml addition solution is pale green, changes to yellow-green (3.00 ml), blue-green (5.0 to 10.0 ml) then blue to blue-purple.

"Only certain amines of the aniline type give the color reaction. Similar aromatic compounds with the nitrogen attached to the benzene nucleus give various colors, depending upon the type of substituents present.... An acetyl group on the nitrogen inhibits color formation. Alkyl groups substituted in the amino group intensify the color. Aryl groups exhibit a similar effect. NH_2 as a ring substituent intensifies the color.... Compounds of the benzidine type and the naphthylamines give deep colors.... The color is specific to the amine and can be produced with all reactive types of clay."

Reactions of Clay Minerals with Benzidine:

Benzidine and benzidine hydrochloride have been used widely in tests to identify clay minerals. Unfortunately, the molecular forms developed by oxidation of benzidine are not understood fully but it is generally supposed that the colored form is a semiquinone, or possibly a meriquinone (1, 27, 30) developed simultaneously with, or following, adsorption of the benzidine or its hydrochloride by the clay crystal.

As has been shown previously, the benzidine is adsorbed by the clay; hence, the coloration is firmly adherent to the particles of clay minerals. If a suspension of treated montmorillonite is allowed to settle, the solution becomes colorless, although the clay remains blue. The coloration cannot be extracted and maintained in solution independently of the clay mineral, but the color of the clay will fade with leaching by a solvent for benzidine. With drying, the color changes through green and yellow-green to greenish yellow if the supply of benzidine is deficient or if the montmorillonite is the calcium type. However, with excess benzidine, Na- and H-montmorillonites remain blue or become blue-gray. With acidification, yellow and yellow-green hues develop. If the benzidine is applied in a solution of absolute alcohol, samples of montmorillonite which become purple-blue in aqueous solution do not change color or become yellow- green or blue-green; but all become yellow-green or greenish yellow upon drying. Water added at any stage of the alcohol treatment develops the typical blue-purple blue.

Treatment of montmorillonite with reducing agents, such as $SnCl_2$, prevents the color-producing reaction but the color-producing capacity in the oxidation-reduction reaction can be restored by an oxidizing agent (1). Prior treatment of a clay with organic compounds which are adsorbed in preference to benzidine will inhibit color development (27). Consequently, not only oxidizing ions but also adsorption of the benzidine by the clay mineral, bringing the amino groups into close proximity to the oxidizing centers in the clay lattice, is necessary to the characteristic color reaction.

The effect of changing acidity of benzidine-treated montmorillonite upon the color of the clay is significant. Acidification of a dilute slurry of Na-montmorillonite, Osage, Wyo. with 0.05 *N* HCl reveals color changes as follows:

pH	Color
10.8	Purple-blue fades to gray
10.8–3.0	Purple-blue to blue
3.0–2.3	Lavender
2.3–0.0	Yellow

Hauser and Leggett (28) conclude tentatively that the color is determined by the amount of acid salts formed with the amino groups, and the resulting resonance within the oxidized benzidine molecule. They state:

> "In basic media no salt formation occurs and the resonance goes through the two rings of the biphenyl nucleus, resulting in a deep blue. If sufficient acid is added to form salt with one-half the amine present, the resonance is restricted to one ring, and ... the yellow color typical of imine compounds is produced. More acid forms salt with all of the amino groups, no resonance is induced, and a colorless state results."

More recently, Hauser and LeBeau (29) attribute the color change to release from the clay crystal of atomic oxygen.

Identification of Clay Minerals by Reaction with Benzidine:

Because of the interference with color reactions of benzidine by organic matter and inorganic cations and by the varying acidity of natural soils, the benzidine test for montmorillonite clays has been criticized severely (31, 32, 33, 34, 35). Page (31) concluded that ferric compounds present in soils will cause a positive color reaction with the hydrochloride, even in the absence of montmorillonite; but experience of Hendricks and Alexander (27) and others (3), including the present authors, does not demonstrate interference, especially if aqueous solution of benzidine (rather than of the hydrochloride) is used.

Nevertheless, the benzidine test described by Hendricks and Alexander (27), is unsatisfactory as a generally applicable technique. Montmorillonitic soils frequently do not produce characteristic colors, and others produce various shades of blue-green, pale bluish-gray, green, or yellow. Recommended procedures to improve reliability of the test by drying of the sample (33) or application of a solution of ferric chloride (34) have not been successful. Moreover, some montmorillonite-free soils, particularly illitic types and some vermiculites, yield blue or blue-purple color. However, a sufficiently large proportion of montmorillonitic soils yield characteristic colors to maintain interest in the method. Tests of randomly selected samples of soils, sediments, and rocks gave correct indications of the presence or absence of montmorillonite-type minerals in only 21 of 28. In another series of tests (36) correct indications were 53 of 63.

Reliability of the method is improved considerably if organic matter and soluble compounds are removed prior to the test and if the pH is controlled. If randomly selected soils are treated to remove acid-soluble substances and organic matter, entirely satisfactory results are achieved with addition of an excess of aqueous benzidine solution and sufficient hydrochloric acid to reduce the pH of

TABLE IV.—SUMMARY OF BENZIDINE TEST WITH CLAY AND CLAYLIKE MINERALS.[a]

Clay Mineral	Number of Samples Tested	Original Color[b]	Original Sample[b] (Wet)	Acid-Treated Sample with Benzidine[c] (Wet)	Acid-Treated Sample with Excess Benzidine pH 1[d] (Wet)	Acid-Treated Sample with Excess Benzidine pH 1[e] (Dry)
Kaolinite	11	N 10 to N 2.5	N 10 to 5 B 8/2	N 10 to 5 B 8/2	N 10 to 10 Y 9/2	Colorless
Anauxite	1	N 10	5 B 9/1	5 B 8/4	N 10	Colorless
Halloysite	3	N 10 to 5 B 9/2	N 10 to 5 B 8/2	N 10 to 5 B 8/2	N 10	Colorless
Nacrite and dickite	3	N 10 to 10 BG 9/2	N 10	N 10 to 5 B 8/2	N 10	Colorless
Montmorillonite	11	N 9 to 10 Y 8/2	5 PB 3/10–5/10 to 10 B 6/6	5 PB 3/12 to 10 B 5/6	10 Y 5/8–8/4 to 5 GY 7/8	10 Y 8/8–5/6
Nontronite	1	10 Y 8/6	10 BG 5/6	10 B 3/8	10 Y 6/6	10 Y 7/8
Hectorite	1	N 10	5 PB around edges of test mass	N 10	10 Y 8/6	10 Y 8/6
Illite	2	N 5 to 10 GY 5/4	10 B 3/6 to 5 PB 5/10	5 PB 4/10[f]	N 5 to 5 GY 4/4	5 GY 7/10[g]
Glauconite	1	10 GY 5/6	10 B 3/6	10 B 3/6	10 GY 5/6	10 Y 4/8[g]
Celadonite	1	10 G 5/6	5 BG 4/6	5 BG 4/6	10 G 5/6	5 GY 4/4–6/4[g]
Vermiculite	3	10 Y 6/2–7/4	10 GY 5/2–10 GY 8/4–5 B 6/4	5 B 4/6–5 BG 5/2	10 Y 6/2–7/4	10 Y 6/2–7/4[g]
Pinite	1	5 G 8/4	5 BG 6/6–5/6	5 PB 3/8	5 G 8/4	Colorless
Sericite	1	N 10	N 10	N 10	N 10	Colorless
Talc	1	N 10	N 10	N 10	N 10	Colorless
Pyrophyllite	1	N 10	N 10	N 10	N 10	Colorless
Palygorskite	1	N 10	N 10	N 10	N 10	Colorless

[a] Procedure 1, Appendix IV, colors according to Munsell system.
[b] Reflected light, see Appendix I.
[c] Reflected light, 5–10 min. after addition of benzidine solution.
[d] Reflected light, 4 hr after acidification.
[e] Transmitted light under microscope.
[f] Not all illites become blue in benzidine solution.
[g] Body color.

TABLE V.—CHANGE OF pH OF ACID-TREATED CLAYS WITH ADDITION OF WATER.[a]

Distilled water added, ml	pH of Mixture								
	0.00	0.75	1.00	1.50	3.00	10.00	50.00	100.00	200.00
Kaolinite									
Mesa Alta, New Mexico (API No. 9a)	5.2	5.8	5.2	5.1	5.0	4.8	4.2	4.2	4.3
Halloysite									
Spruce Pine, North Carolina (API No. 51)	1.3	3.8	3.7	3.8	3.8	4.0	4.2	4.2	4.2
Illite									
Fithian, Illinois (API No. 35)	3.7	4.6	4.2	3.9	3.6	4.0	4.2	4.4	4.5
Montmorillonite									
Clay Spur, Wyoming (API No. 26)	0.6	1.5	1.4	1.7	2.1	2.4	3.6	3.9	4.1
Hectorite									
Hector, California (API No. 34d)	5.5	6.6	6.6	6.6	6.8	7.2	7.1	7.1	7.2

[a] 2.000 g room-dry sample crushed to pass the No. 200 sieve. Clay packed dry about glass electrodes of Beckman pH-meter for initial determination; readings are variable depending on packing of sample, being lower for greater compaction. Water added from buret, and mixed thoroughly, readings taken by embedding or immersing electrodes in the mixture. Determinations on slurries made during agitation.

[b] Sample selected from reference clays, Project 49, American Petroleum Institute (39).

the slurry to approximately 1 (Table IV).

Note that hectorite, Hector, Calif., which did not develop the characteristic blue or purple-blue oxidation form, nevertheless produced the greenish-yellow hue upon acidification and drying. Similarly, montmorillonites from Otay, Calif., and Santa Rita, N. Mex., which produced no color reaction with benzidine added to the untreated sample, produce the characteristic greenish yellow in this procedure.

Reference to the criteria for evaluating staining tests[3a] indicates that the benzidine test applied to acid-treated materials meets all the requirements satisfactorily. Further research might improve distinction between minerals, although probably not without sacrifice in criteria 2 and 4.

[3a] See p. 1216.

STAINING INVOLVING ACID-BASE REACTIONS

Interaction of Acid-Treated Clay Minerals with Acid-base Indicators:

If natural clays are washed with sufficient aqueous solution of hydrochloric or other acid, original exchangeable cations will be replaced by H^+ ions, and even when the excess acid is removed by repeated washing, an aqueous slurry of the clay will be slightly or strongly acidic, depending in large part upon the amount of clay present and its base exchange capacity. Clays so treated are weak acids, releasing ionized H^+ into the free water but retaining sufficient H^+ ions in close proximity to the clay particles to produce a more strongly acidic condition in adsorbed water.

If certain coal-tar dyes are added to the slurry, molecules of the dye will be adsorbed by the clay crystals. Furthermore, if the dye characteristically develops certain colors under moderately or highly acidic conditions, these colors will be developed in the adsorbed water film. If the pH value of the free water lies below the transition interval for the color of the dye and the pH of the adsorbed water lies above the interval, the clay crystal, or aggregate of crystals, will stain a color different from that of the solution as a whole and different from that of particles which passively adsorb the solution or merely absorb the dye.

From this discussion, it will be seen that development of a satisfactory staining test, based upon acid-base reactions, involves selection of dyes which show characteristic colors in response to the pH value developed in water films adsorbed by the various species of acid-treated clays (25), and these color reactions should be subject to a minimum of interference from factors not inherent in the clay minerals.

The acidity of mixtures of acid-treated clay and water can be determined by simple experiments (Table V). The pH of a room-dry acid-treated Wyoming bentonite was found to be 0.60 and probably would be found to be lower if the clay were packed more tightly about the electrodes. With the addition of water, the acidity decreases rapidly. The pH value of room-dry kaolinite, hectorite, halloysite, and illite ranges from 1.3 to 5.5, and the pH value of the aqueous slurry ranges from 4.2 to 7.2.

Dyes and Solvents for Use in Acid-Base Reaction of Clay Minerals:

Stains most commonly used in acid-base reactions for identification of clay minerals are triphenylmethane, azo, and azine dyes. From these and other groups of aniline dyes, compounds undergoing color change over the whole range of acidity may be selected. In the present study, ten dyes, changing color to a pronounced degree in the range of pH from 5.0 to 0.0, were employed.

Various solvents are available as carriers for the dyes, the choice depending largely upon the influence of the solvent upon the color reactions, solubility of the dyes, penetrating power, magnitude and constancy of refractive index, and volatility. For clay mineral identification, solvents whose refractive index lies in the range 1.52 to 1.60 are most convenient. Aniline (n = 1.5863 at 20.0 C) with isoamyl isovalerate to lower the index (37) and a mixture of quinoline and paraldehyde (38) have been used as solvents in microscopy of clays. Unfortunately, the refractive index of the latter increases rapidly on exposure and aniline takes part in oxidation-reduction reactions with montmorillonite minerals.

Nitrobenzene is recommended as a dye solvent by Faust (25) because of its high penetrating power and because its refractive index (n = 1.5524 at 20.0 C) approaches ϵ of quartz, and thus facilitates recognition of this common constituent of soils. Moreover, its refractive index does not change with exposure upon a microscopical slide. Nitrobenzene does not interfere with acid-base reactions between clays and dyes.

In the Petrographic Laboratory, nitrobenzene is used most commonly as a dye solvent in the staining procedures dependent upon acid-base reactions, safranine Y and malachite green being the most discriminating stains.[3] Addition of the dyes to nitrobenzene does not change the refractive index more than ±0.0002. The saturated nitrobenzene solutions contain 339 mg per liter of safranine Y and 1,935 mg per liter of malachite green. Because of the widely different solubilities, malachite green develops the stronger color in clay particles. The nitrobenzene solutions show pronounced absorption maxima, centering at 600 mμ and 525 mμ for malachite green and safranine Y, respectively.

Results of Acid-Base Reactions with Aqueous Solutions of Dyes:

To establish the staining reactions upon the common clay minerals, saturated aqueous solutions were prepared with five aniline dyes which show marked change of color in ranges of pH between 0.0 and 5.0. The pH value of the dye solutions was adjusted at approximately 3, 7, and 10, with aqueous solutions of NH_4OH and HCl. The samples of clay were selected from the clay mineral standards, Project 49, American Petroleum Institute (**39**), and treated to remove iron oxides and carbonates and to produce clay minerals in which hydrogen is the exchangeable cation.[4] With bromphenol blue, various hues of yellow are developed at pH 3.0 and 7.0; but diagnostic differences between the clays are not apparent. At pH 9.7 differences in hue are not satisfactory in aqueous solution, except for a distinctive purple developed in halloysite. Only slight differences in hue are produced in the clays by alizarin red S in aqueous solution, and addition of nitrobenzene changes the colors within the clay minerals to a narrow range of hues. Thymol blue permits easy distinction only of montmorillonite, except after drying of the mounts from the solution at pH 7.0. In the latter treatment, montmorillonite is a distinctive purple-red purple; illite is red; halloysite is yellow-red yellow; and kaolinite is yellow-red.

Most satisfactory of all the stains in this procedure are safranine Y and malachite green. With safranine Y in aqueous solution, the distinction between kaolinite and halloysite, or between illite and montmorillonite is not dependable. However, characteristic colors are developed with drying of the aqueous solutions of pH 3.32 and 7.05, and these colors are deepened with the addition of nitrobenzene. With application of the dye solution at pH 9.71, distinction between kaolinite and halloysite is not dependable.

With malachite green, results analogous to the safranine Y are obtained, except in hues of red, yellow, green, and blue. At pH 9.7 the dye solution is almost colorless, and distinctive colors are not produced until addition of nitrobenzene after drying of the aqueous solution; colors of illite and montmorillonite are faint, but those of kaolinite and halloysite are strong and diagnostic.

Most impressive in the application of the safranine Y and malachite green solution is the development of the strong, purple-blue, or red or yellow-red in montmorillonite with drying. Nontronite stains as does montmorillonite in these tests. However, hectorite becomes red-purple red with addition of nitrobenzene after treatment with the aqueous safranine Y solution, and yellow-red yellow to green-yellow with addition of nitrobenzene after treatment with the malachite green solution.

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[3] Safranine Y and malachite green used in the reported tests are produced as biological stains by The Coleman and Bell Company, Norwood, Ohio, and are marketed as the chloride and oxalate, respectively, under Color Index Numbers 657 and 841.

[4] In each test a small portion (about 50 mg) of the acid-treated sample was placed upon a glass microscopical slide; one drop of the selected dye solution was added and mixed thoroughly with the clay. After 5 min., the color of the clay was observed and recorded. The mount then was dried in the warm air rising from the microscope lamp (at about 55 C), whereupon two drops of nitrobenzene were added, the precipitated dye was dissolved by stirring, and the color of the minerals was observed and recorded at 2 min following addition of the nitrobenzene.

With alizarin red S, brom phenol blue, and thymol blue, the acid colors develop at pH less than 1.2 to 3.8; it will be noted that only minor changes occur with drying in the hue developed in H-montmorillonite by the aqueous solution of these dyes. However, colors characteristic of the most acid range for safranine Y and malachite green develop only at pH less than 0.3 and 0.0, respectively. Evidently, this pH is not attained in adsorbed water films unless they are reduced in volume by drying at relatively low humidity, for the montmorillonite stains red-purple in aqueous safranine Y solutions and green to yellow in aqueous malachite green solutions, becoming purple-blue and red to yellow-red, respectively, only with drying.

Results of Acid-Base Reactions with Nitrobenzene Solutions of Dyes:

Nine dyes in saturated nitrobenzene solution were applied to acid-treated portions of the selected samples of clay minerals.[5] The results indicate that alizarin red S and congo red 4B are unsatisfactory stains because of low intensity of color development in most applications, and methylene blue does not develop colors distinctive of any of the clay minerals. Crystal violet, thymol blue, fuchsin basic, and methylene violet develop colors permitting distinction of montmorillonite from kaolinite, illite, and halloysite, but the latter three minerals cannot be separated dependably from one another.

Both malachite green and safranine Y in nitrobenzene permit dependable separation of the four minerals from each other. The colors developed in the clay minerals following application of these dyes in nitrobenzene are similar to those developed on application of nitrobenzene to the dried samples previously treated with aqueous solutions at pH near 3 and 7.

Identification of Clay Minerals by Reaction with Malachite Green and Safranine Y:

Because of the general superiority of malachite green and safranine Y over other dyes which have been applied in the staining tests, these two dyes have been used routinely in microscopy of soils and rocks in the Petrographic Laboratory since 1941 (36). Color reactions characteristic of various clay minerals and their common associates are summarized in Table VI.

Caution must be used in addition of the dye solutions, inasmuch as a great excess of the dyes leads to saturation of clay particles with the dye, causing production of red-purple red and blue-green blue hues with safranine Y and malachite green, respectively, even in H-montmorillonite clays. As has been demonstrated previously, adsorption of the dyes by acid-treated clays is accompanied by release of adsorbed H^+ ions. Consequently, excessive adsorption of the dyes leads to decrease in acidity of the clay particles sufficient to prevent the characteristic color transformations.

With addition of saturated nitrobenzene solutions of safranine Y and malachite green to acid-treated montmorillonite in a range of sizes, a virtually instantaneous reaction converts the large particles (greater than 50 μ) to blue-purple or yellow-red, respectively, and the small particles (less than 5 μ) to red-purple red or blue-green. Intermediate sizes are intermediate in color or show rims of red-purple red or blue-green. If the dye in solution is depleted by the adsorption reaction, the red-purple red or blue-green stains gradually grade over to blue-purple or yellow-red. However, if the dye in solution is in excess, the red-purple red or blue-green remains in the smallest particles; or, indeed, rims of these hues may form on the larger particles.

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[5] The dye solution was added slowly to a portion of the acid-treated sample, with intermittent stirring, until sufficient solution was present to permit microscopical examination. Color in the clay mineral particles was observed at 2 and 10 min after addition of the dye solution.

TABLE VI.—SUMMARY OF ACID-BASE REACTIONS WITH ACID-TREATED CLAY AND CLAYLIKE MINERALS.[a]

Clay Minera	Number of Samples Tested	Original Color	Color with Safranine Y in Nitrobenzene	Color with Malachite Green in Nitrobenzene
Kaolinite	21	N 10 to 2.5	5 R 4/10–10 RP 6/8[b]	5 B 6/6–10 G 6/6[b]
Anauxite	1	Colorless	5 R 6/10–5 RP 6/12[b]	5 BG 6/10–10 G 6/10[b]
Halloysite	5	N 10 to 5 B 9/2	5 R 5/10–5 PB 5/10[c]	5 B 6/6–10 Y 6/10[c]
Nacrite	1	N 10	10 RP 5/10 to colorless	5 B 5/6 to colorless
Dickite	6	N 10 to 10 BG 9/2	10 RP 6/10–5 R 5/10 to colorless	5 B 7/6–5 BG 5/6 to colorless
Montmorillonite	15	N 10 to 10 Y 8/6	10 PB 5/10–5 PB 5/10	10 YR 6/6–5 YR 7/10
Nontronite	1	10 Y 8/6	5 PB 5/10–7/10	5 R 6/10–3/10
Hectorite	1	N 10	10 RP 5/12–7/10	10 YR 8/10–5 GY 4/10
Illite	2	N 5 and 5 GY 7/2	10 RP 5/10–10 P 5/10	5 BG 5/6–5 GY 5/8
Glauconite	1	10 Y 4/8	10 Y 3/2 to 5 YR 5/10[d]	5 GY 3/4–6/4[d]
Celadonite	1	5 GY 4/4 to 10 Y 7/8	10 RP 6/10 to 10 GY 4/4[d]	10 Y 5/6 to 5 GY 5/8[d]
Vermiculite	2	6 Y 4/12–9/12	5 R 6/12 to 10 P 7/10	5 BG 6/10 to 8 YR 7/10[d]
Pinite	1	2 Y 9/8 to N 10	8 RP 5/10–8/10	5 BG 5/10–8/10
Sericite	1	Colorless	Colorless	Colorless
Talc	1	Colorless	Colorless	Colorless
Pyrophyllite	1	Colorless	Colorless	Colorless
Palygorskite (attapulgite)	4	N 10	5 RP 5/10 to 10 RP 5/10	5 BG 5/6–10 BG 5/6

[a] See Appendix III for procedure; colors designated according to Munsell System (Appendix I).
[b] Moderate pleochroism.
[c] Mottled.
[d] Modified by body color.

Experiments with synthetic mixtures of quartz and Wyoming bentonite crushed to pass the No. 200 sieve demonstrate that permanent red-purple red hues are developed in acid-treated montmorillonite by saturated nitrobenzene solution of safranine Y if the mixture contains less than about 20 per cent of montmorillonite, and that blue-green hues are developed by saturated nitrobenzene solution of malachite green if montmorillonite constitutes less than about 15 per cent of the mixture. However, if the saturated solutions are diluted by an equal volume of nitrobenzene, the red-purple red and blue-green hues may be avoided by use of a minimum of dye solution on samples containing as little as 5 per cent of montmorillonite.

Safranine Y develops a red-purple red far more readily than malachite green develops blue-green colors in the acid-treated montmorillonite. Using a sample containing 15 per cent by weight of acid-treated bentonite, twice the volume of 1 to 1 mixture of saturated solution of malachite green in nitrobenzene was required to produce oversaturation of the clay as was required of a similarly prepared solution of safranine Y.

Acid-treated illite resists oversaturation and neutralization or exchange of the hydrogen ions, but with application of the safranine Y solution in great excess, hues of red-purple develop, thus possibly permitting confusion of illite with kaolinite minerals. However, saturated nitrobenzene solutions of safranine Y and of malachite green can be applied safely even if illite constitutes as little as 5 per cent of the sample.

In selecting the proper concentration of dye and the amount of dye solution to apply to the mount, a balance must be achieved between a sufficiency of dye to produce stains of satisfactory intensity and an excess of dye such as might produce anomalous results.

Reference to the criteria for evaluating staining tests, shows that the tests with safranine Y and malachite green are superior to the test with benzidine as described in Appendix IV, so far as preliminary treatment (criterion 2)[5a] is concerned, but that possible oversaturation of clay particles with safranine Y and malachite green weakens these tests according to criterion 4. Malachite green is superior to safranine Y under criterion 4; but when weakly developed, the yellow and green hues of malachite green are not always readily distinguished from original body color (criterion 1b).[5a]

SUMMARY OF STAINING REACTIONS FOR CLAY AND CLAYLIKE MINERALS

Kaolinite Group:

Kaolinite merely absorbs and adsorbs safranine Y and malachite green, becoming red to red-purple red or blue to green-blue green with these dyes, respectively. Pleochroism is induced by adsorption of these dyes, but the intensity is influenced by the degree of crystallinity. Well-formed, vermicular crystals are markedly pleochroic. The characteristic pleochroic scheme is blue and yellowish-red perpendicular to the cleavage, and green-yellow green and reddish-purple parallel to the cleavage, with malachite green and safranine Y, respectively. No characteristic color reaction takes place with benzidine.

Anauxite is similar to kaolinite in its reaction with safranine Y and malachite green except that the pleochroism is blue-green and red perpendicular to the cleavage, and green-blue green and red-purple parallel to the cleavage with these dyes, respectively. These differences between kaolinite and anauxite probably will not be reliable means of separation. No characteristic color reaction takes place between benzidine and acid-treated or untreated anauxite, if the mixture is acidified immediately to pH 1. To distinguish between anauxite and kaolinite, apply the benzidine solution to acid-treated material and dry at room temperature. Then add a drop of 1:1 hydrochloric acid and dry at room temperature. Under the microscope in transmitted light, kaolinite is colorless whereas anauxite is yellow (10 Y).

Single crystals of dickite do not adsorb malachite green and safranine Y; but aggregated masses of minute crystals become blue to blue green and red-purple to red with these dyes, respectively. Induced pleochroism is very weak; the scheme is light blue and yellow red perpendicular to the cleavage and yellowish-green to colorless and light reddish purple parallel to the cleavage with malachite green and safranine Y, respectively. Dickite causes no color changes with benzidine, if the solution is added to untreated material, or if the mixture of acid-treated sample and solution is acidified immediately to pH 1.

Nacrite in large crystals does not adsorb the dyes readily, but small fragments stain in a blotchy manner. Pleochroism is faint, being yellowish-green to blue in malachite green solution and red-purple red to yellowish-red in safranine Y solution. Nacrite reacts as does dickite in tests with benzidine solution.

Halloysite Group:

Halloysite in original condition characteristically adsorbs and absorbs malachite green and safranine Y, becoming blue-green and red to red-purple. No pleochroism is evident in the halloysites examined. After acid treatment, halloysite particles typically develop an inner green-yellow or purple zone, with either a blue-green or red-purple red outer zone with these dyes. These colors in the inner zone usually fade within ½ hr to blue-green or red-purple red. This anomalous staining can be prevented by boiling the thoroughly washed sample, and so probably is caused by excess acid tenaciously retained by halloysite crystals. Some naturally acidic halloysites, such as those from Eureka, Utah, and Cadouin, France (36), react similarly without acid treatment. In practice, these anomalous colors are aids in distinguishing members of the kaolinite group from the halloysites. No characteristic color reaction occurs between benzidine and untreated halloysites, or between acid-treated halloysites and benzidine immediately acidified to pH 1.

[5a] See p. 1216.

Montmorillonite Group:

Acid-treated montmorillonite and nontronite develop characteristic yellow-red or red and purple-blue purple hues with malachite green and safranine Y, respectively. Acid-treated hectorite merely adsorbs the dyes without changing their color. Nontronite and hectorite are easily decomposed by acid, so that caution is required in acid treatment. No data are available on saponite. Montmorillonite and nontronite characteristically become purple-blue purple with benzidine; some montmorillonite minerals and hectorite containing less than 1 per cent of ferric iron do not cause development of these colors. With acidification to pH 1 of the benzidine solution-sample mixture, any of these clays develops a characteristic greenish-yellow.

Micaceous Clays and Claylike Minerals:

Acid-treated illite and certain vermiculites become purple-red purple to red in safranine Y and yellow-red to blue-green with malachite green. With addition of benzidine to the untreated sample, some illites (36) and some vermiculites (42) develop hues of blue. However, if the benzidine solution is added to acid-treated vermiculite, and the mixture immediately acidified to pH 1, the original blue disappears, thus permitting distinction of these minerals from montmorillonite minerals, which characteristically become and remain greenish yellow with acidification and drying. Illite and adsorptive vermiculites are indistinguishable in either acid-base oroxidation reduction reactions, so far as these techniques have been developed. With gradation of illite to better crystallized, less adsorptive forms, such as pinite and sericite, intensity of color development and degree of color change in the adsorbed dyes decrease (Table VI).

Glauconite and celadonite likewise cannot be distinguished by the staining tests, but their original body colors are sufficiently characteristic to differentiate them from other micaceous clay and claylike minerals.

Palygorskite Group:

Palygorskite (attapulgite) merely adsorbs and absorbs the safranine Y and malachite green, becoming red-purple to red-purple red or blue-green to blue-green blue, with or without prior acid treatment. This mineral does not change color in any of the tests with benzidine, although small amounts of admixed montmorillonite may change the appearance of the sample as a whole. This characteristic lack of stain reaction, high birefringence, low index of refraction, and common habit in hand specimen permit distinction of palygorskite from other common clay and claylike minerals.

CLAY STAINING TESTS IN ANALYSIS OF SOILS AND ROCKS

Analysis of clay mineral content of soils and rocks in the Petrographic Laboratory is based upon the application of a nitrobenzene solution of safranine Y and malachite green and aqueous solutions of benzidine to acid-treated samples. The benzidine test for montmorillonite-type clays can be applied to untreated soils when preliminary work demonstrates its validity in the suite of materials under investigation.

The acid-base reactions are best suited to analysis if proportions of montmorillonite clays are moderate to high, if distinction between kaolinite group minerals or between halloysites and kaolinite group minerals is necessary, and if rapid determinations are essential. The benzidine test of acid-treated samples (Procedure 1, Appendix IV) is especially suited to analysis of samples containing small proportions of adsorptive clays, or if distinction between montmorillonite-type minerals and micaceous clays and claylike minerals is required. Procedure 2, Appendix IV, is designed to separate kaolinite and anauxites.

Staining tests for clay mineral identification can be used alone with other microscopical techniques or to supplement analysis of suites of samples when other methods, such as X-ray diffraction and differential thermal methods, are available. Qualitative identification of major clay components of samples usually can be made by staining tests without the microscope, using either the oxidation-reduction reactions or acid-base reactions. Selection of representative portions of the sample for mounting and accuracy of the determinations are increased considerably by initial sizing of the material. Synthetic mixtures of clay and nonclay minerals in varying proportions and size fractions permit checking of determinations made on test samples. Various types of ruled eye-piece micrometers are available commercially or may be prepared (**40, 41**) to aid in determination of relative areas occupied in the mount by the several ingredients.

CONCLUSION

When used as stains in microscopical analysis of soils and rocks, safranine Y and malachite green in nitrobenzene solution produce characteristic colors in most montmorillonite-type minerals (all except hectorite), halloysites, and micaceous clays and claylike minerals, such as illite and certain vermiculites, after acid treatment of the samples. Kaolinite and anauxite develop moderate pleochroism, whereas pleochroism is weak or absent in dickite and nacrite. Newly developed techniques of staining with benzidine involving acidification of the sample and control of the pH of the mixture will identify even minute quantites of montmorillonite-type minerals (including hectorite) in soils, and readily differentiate them from illite and vermiculite, palygorskite (attapulgite), the halloysites, and kaolinite minerals. Moreover, kaolinite can be distinguished from anauxite.

Staining of acid-treated soils and rocks with safranine Y and malachite green or with benzidine are complementary techniques. The methods are reliable and applicable generally in microscopical analysis of soils and rocks.

Further work undoubtedly will develop staining methods capable of more precise identification of clay and claylike minerals.

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APPENDIX I

The Munsell Color System

The Munsell system of color notation describes colors according to hue, value, and chroma. The concept may be visualized as based upon a hypothetical color cylinder constructed with a vertical neutral axis grading from white at the top to black at the bottom and divided into nine degrees of value. Around the circumference are distributed the ten major hues, each being indicated by one or two letters and divided into ten numerical divisions such that 5 marks the middle of a hue and 10 the boundary between hues. Thus any hue may be designated by appropriate number and letters, as 5 RP (red-purple), or 10 BG (blue-green blue). From the central axis outwardly, each radius is divided into 16 steps of chroma (degrees of saturation). Thus any radial plane bounded by the central axis and the periphery of the cylinder constitutes a color chart for a particular hue, the colors grading from dark at the bottom to light at the top and from gray adjacent to the axis to the most vivid colors of the hue at the outer edge. Colors whose chroma is 3 or less are drab, and those whose value is 9 or more are very pale; these are the colors commonly found in natural materials.

According to this scheme, any color can be identified by designation of the hue, value, and chroma. In the notation of color, the symbol for hue is written first, followed by a symbol in fractional form in which the numerator indicates the value and the denominator represents chroma; for example, if 10 yellow is the hue, 5 the value, and 6 the chroma, the notation is 10 Y 5/6.

Hues are designated as follows:

N—neutral white, gray and black
B—Blue
PB—Purple-blue
P—Purple
RP—Red-purple
R—Red
YR—Yellow-red
Y-Yellow
GY-Green-yellow
G—Green
BG—Blue-green

APPENDIX II

Acid Treatment of Samples

Statement:

Staining tests may be applied to an entire soil or to individual size fractions obtained by mechanical analysis, as in accordance with ASTM Method D422-39.[6] If excessive amounts of iron oxides or organic matter are present, they should be removed to facilitate microscopical examination (**43, 44, 45**). All methods of sample treatment not necessary to efficient and reliable microscopy should be avoided, mainly because crystals of clay minerals are easily disintegrated or decomposed. All samples should be examined microscopically before treatment to detect original body color of the constituents and to afford a check upon possible alteration during subsequent treatment.

Procedure:

To acid-treat the sample or selected fractions, place approximately 20 g of the material in a beaker and add 200 ml of 1 to 25 hydrochloric acid (1 part concentrated hydrochloric acid to 25 parts of distilled water). If carbonates are present, increase the amount of acid proportionately.[7]

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[6] Method of Mechanical Analysis of Soils (D422-39), 1949 Book of ASTM Standards, Part 3, p. 1162.

[7] Hydrochloric acid as strong as 1 to 1 may be used for soils containing members of the kaolinite group, the halloysites, montmorillonite, beidellite, illite, celadonite, glauconite, or palygorskite. But hectorite and some nontronites and vermiculites are destroyed by strong acid solutions. Dilution of concentrated hydrochloric acid to 1 to 25 is required to prevent decomposition of hectorite, whereas dilution to 1 to 10 will suffice for nontronite and vermiculite. Hydrochloric acid diluted with 1 part or less of distilled water is quite effective in dissolving iron oxides from soils, but dilutions as great as 1 to 10 are comparatively ineffective. Experience with soils in specific areas or prior knowledge from other work may indicate that the higher concentrations of hydrochloric acid may be used without damage to the soil constituents; their use will facilitate acidification and cleaning.

Stir or agitate the mixture frequently for ½ hr; pour off the supernatant solution after settling of the sample or centrifuging. Repeat the acidification of the sample as before using two additional 200 ml charges of 1 to 25 hydrochloric acid. Wash the sample repeatedly using about 500 ml of distilled water for each washing. To expedite, decant only the supernatant liquid with a minimum of fines into a filter paper, retaining the bulk of the sample in the beaker. Continue washing until no silver chloride precipitate is observed in the filtrate upon addition of silver nitrate solution.

Dry the sample and the filter paper with retained fines at 105 C for 24 hr (or to constant weight). Remove the fines from the filter paper and mix them thoroughly in a mortar into the sample retained in the beaker.[8]

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[8] If only a very small proportion of fines is present in the sample, qualitative observation of clay minerals is facilitated if the fines are examined directly in the staining tests.

APPENDIX III

STAINING TEST FOR CLAY MINERAL IDENTIFICATION USING SAFRANINE Y AND MALACHITE GREEN

Staining agents used are saturated nitrobenzene solutions of malachite green ($2C_{23}H_{25}N_2 \cdot 3C_2H_2O_4$) and of safranine Y (a mixture of $C_{21}H_{21}N_4Cl$ and $C_{20}H_{19}N_4Cl$)[3] and solutions of these dyes prepared by diluting the saturated solutions with an equal volume of pure nitrobenzene.

To perform the test, place a small portion (about 50 mg) of pulverized or sized, acid-treated sample upon each of two glass microscopical slides. Add a drop of the diluted nitrobenzene solution of safranine Y to the mount and mix thoroughly; add successive aliquots of the dye solution drop by drop with intermittent mixing until just sufficient liquid has been added to produce a uniform film on the slide. Place a cover slip over the mount. Begin the microscopical examination in transmitted light under the petrographic microscope about 2 min after the first addition of dye solution. Repeat preparation of the second mount using the diluted solution of malachite green. If color development in montmorillonite-type minerals is faint, repeat preparation of the mounts with the saturated solutions of the dyes.

It will be noted (Fig. 1) that the benzidine test is to be applied if separation of illite and vermiculite from hectorite, palygorskite, and traces of montmorillonite-type minerals or of anauxite from kaolinite, is to be accomplished by microscopical methods.

Characteristic staining reactions of common clay and claylike minerals are indicated in Table VI. Note that the acid-base reaction is unreliable for identification of montmorillonite-type minerals if they constitute less than 5 per cent of the sample.

APPENDIX IV

STAINING TEST FOR CLAY MINERAL IDENTIFICATION USING BENZIDINE

Prepare the sample as indicated in Appendix II.

Procedure 1:

By preliminary microscopical examination, manipulation of the moistened sample, or inspection of mechanical analysis data, roughly estimate the clay content of the material. If the estimated clay content is less than 50 per cent, use Procedure 1*a* in the staining test; if it exceeds 50 per cent or if no reliable estimate is possible, use Procedure 1*b*.[9]

Procedure 1(a):

Place a small quantity (about. 015 g) of pulverized, prepared sample in one depression of a spot plate, and add saturated water solution of benzidine drop by drop mixing continuously into the sample, until the mixture almost fills the depression. Add 1 to 1 hydrochloric acid, mixing thoroughly, until the pH of the mixture is reduced to about 1.[10]

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[9] This initial separation of samples is designated because success of the staining procedure requires that sufficient benzidine be available to the clay mineral constituents to effect satisfactory color change. Consequently, in view of the very low solubility of benzidine in water at room temperature, comparatively large volumes of the solution must be available to samples containing high proportions of adsorptive clay minerals. Procedures 1*a* and 1*b* differ essentially only in the volumes of benzidine solution placed in contact with the sample, and in the time and temperature of contact after acidification.

Allow the mixture to dry in air at room temperature. Place a portion of the treated sample on a glass microscopical slide, add immersion oil ($n = 1.560$), cover the mount with a glass cover slip, and examine under the petrographic microscope in transmitted light.

Results of the test for common clay and claylike minerals are summarized in Table IV and Fig. 1.

Procedure 1(b):

Place a portion of the prepared sample in a 50 ml test tube in sufficient quantity to fill the rounded portion at the bottom. Add saturated water solution of benzidine in an amount equivalent to about five times the volume of the sample, and mix thoroughly. Add 1 to 1 hydrochloric acid, with continuous mixing, until the pH is about 1, as indicated by thymol blue.[10]

Heat the test tube and contents to 90 to 95 C by immersing in a water bath held near boiling; continue this treatment for ½ hr. Permit the sample to settle (or centrifuge using a glass tube), and pour off the supernatant liquid. Remove the sample and allow it to dry at room temperature on a watch glass.

When the sample is dry, proceed with the microscopical examination as outlined in Procedure 1(*a*).

Procedure 2:

If tests with safranine Y or malachite green, or other methods, indicate that kaolinite or anauxite is present, the identity of the mineral or relative proportions of each can be established as follows:

Place a small quantity (about 0.15 g) of prepared, pulverized sample in one depression of a spot plate and add sufficient saturated water solution of benzidine to almost fill the depression. Mix the sample and solution thoroughly, then allow to dry completely in air at room temperature. Add one or two drops of 1 to 1 hydrochloric acid, and allow to dry again at room temperature.

Place a portion of the treated sample on a glass microscopical slide, add immersion oil ($n = 1.560$), cover the mount with a glass cover slip, and examine under the petrographic microscope in transmitted light.

Anauxite stains a characteristic greenish yellow (10Y), whereas kaolinite is colorless (Fig. 1). Note that montmorillonite-type minerals will stain this same hue, but these minerals will have been identified previously in tests with malachite green and safranine Y, or by Procedure 1, above. If montmorillonite-type minerals are present in the same sample with anauxite, anauxite will be identified by its birefringence, index of refraction, or other properties. This distinction will be facilitated by observations made previously in other staining tests in which the montmorillonite-type minerals and anauxite react differently.

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[10] Determine the pH by extracting a drop of liquid from the mixture and adding thymol blue as an indicator. Thymol blue becomes red at pH 1.2.

REFERENCES

(1) H. Weil-Malherbe and J. Weiss, "Colour Reactions and Adsorption of Some Aluminosilicates," *Journal*, Chemical Soc., London, pp. 2164–2169 (1948).

(2) W. H. Cohen and B. H. Knight, "Absorption Phenomena in Soils with Special Reference to their Surface Area," *Journal*, Soc. Chem. Ind., Vol. 66, p. 357–364 (1947).

(3) V. L. Bosazza, "The Adsorption of Some Organic Dyes by Clays and Clay Minerals," *American Mineralogist*, Vol. 29, p. 235–241 (1944).

(4) V. L. Bosazza, "Further Notes on the Adsorption of Malachite Green by Some Clay Minerals," *American Mineralogist*, Vol. 26, p. 396–402 (1941).

(5) V. L. Bosazza, "The Adsorption of Malachite Green by Clays and Allied Minerals," *Nature*, Vol. 146, p. 334 (1940).

(6) V. E. Vedeneeva and M. A. Rateev, "Identification of Disperse Clay Minerals by Dye," *Doklady Akademii Nauk Soyuza S. S. R.*, Vol. 71, p. 141–144 (1950). *Chemical Abstracts*, Vol. 44, p. 9308 (1950).

(7) O. G. Caldwell and C. E. Marshall, "A Study of Some Chemical and Physical Properties of the Clay Minerals Nontronite

Attapulgite, and Saponite," Agricultural Experimental Station, *Research Bull. 354*, University Missouri, p. 27 (1942).

(8) A. N. Winchell, "Studies in the Mica Group," *American Journal of Science*, 5th Series, Vol. 9, p. 309–327 (1925).

(9) R. E. Grim, "Modern Concepts of Clay Minerals," *Journal of Geology*, Vol. 50, p. 225–275 (1942).

(10) P. F. Kerr, "A Decade of Research on the Nature of Clay," *Journal*, Am. Ceramic Soc., Vol. 21, p. 267–286 (1938).

(11) C. S. Ross and S. B. Hendricks, "Minerals of the Montmorillonite Group, Their Origin and Relation to Soils and Clays," U. S. Dept. Interior, Geol. Survey, *Professional Paper No. 205B*, 79 pp. (1945).

(12) G. W. Brindley and K. Robinson, "An X-ray Study of Some Kaolinitic Fireclays," *Transactions*, British Ceramic Soc., Vol. 46, p. 49–62 (1947).

(13) R. E. Grim and R. A. Rowland, "Differential Thermal Analysis of Clay Minerals and Other Hydrous Materials," *American Mineralogist*, Vol. 27, p. 746–761, 801–818 (1942).

(14) S. B. Hendricks and L. T. Alexander, Minerals Present in Soil Colloids," *Soil Science*, Vol. 48, p. 257–271 (1939).

(15) S. B. Hendricks, "Random Structures of Layer Minerals as Illustrated by Cronstedite ($2FeO \cdot Fe_2O_3 \cdot SiO_2 \cdot 2H_2O$); Possible Iron Content of Kaolinite," *American Mineralogist*, Vol. 24, p. 529–539 (1939).

(16) W. F. Bradley, "Diagnostic Criteria for Clay Minerals," *American Mineralogist*, Vol. 30, p. 704–713 (1945).

(17) J. W. Gruner, "The Structure of Vermiculites and Their Collapse by Dehydration," *American Mineralogist*, Vol. 19, p. 557–574 (1934).

(18) S. B. Hendricks and M. E. Jefferson, "Crystal Structure of Vermiculites and Mixed Vermiculite-Chlorites," *American Mineralogist*, Vol. 23, p. 851–862 (1938).

(19) Munsell Color Company, Baltimore, Maryland, "Munsell Book of Color," *Pocket Ed.*, Vol. 1 (1942).

(20) S. B. Hendricks, "Base Exchange of the Clay Mineral Montmorillonite for Organic Cations and Its Dependence on Adsorption Due to Van Der Waals' Forces," *Journal of Physical Chemistry*, Vol. 45, p. 65–81 (1941).

(21) J. E. Gieseking, "Mechanism of Cation Exchange in the Montmorillonite-Beidellite-Nontronite Type of Clay Minerals," *Soil Science*, Vol. 47, p. 1–14 (1939).

(22) W. F. Bradley, "Molecular Associations Between Montmorillonite and Some Polyfunctional Organic Liquids," *Journal*, Am. Chemical Soc., Vol. 67, p. 975–981 (1945).

(23) R. C. Mackenzie, "Complexes of Clays with Organic Compounds. Part II. Investigation of the Ethylene Glycol-Water-Montmorillonite System Using the Karl Fischer Reagent," *Transactions*, Faraday Soc., Vol. 44, p. 368–375 (1948).

(24) C. S. Ross and P. F. Kerr, "The Kaolin Minerals," U. S. Dept. Interior, Geol. Survey, *Professional Paper No. 165*, p. 151–176 (1930).

(25) G. T. Faust, "Staining of Clay Minerals as a Rapid Means of Identification in Natural and Beneficiated Products," U. S. Dept. Interior, Bureau of Mines, *R. I. 3522*, 21 p. (1940).

(26) R. E. Grim, W. H. Allaway, and F. L. Cuthbert, "Reaction of Different Clay Minerals with Some Organic Cations," *Journal*, Am. Ceramic Soc., Vol. 30, p. 137–142 (1947).

(27) S. B. Hendricks and L. T. Alexander, "A Qualitative Color Test for the Montmorillonite Type of Clay Minerals," *Journal*, Am. Soc. Agronomy, Vol. 32, p. 455–458 (1940).

(28) E. A. Hauser and M. B. Leggett, "Color Reactions Between Clays and Amines," *Journal*, Am. Chem. Soc., Vol. 62, p. 1811–1814 (1940).

(29) E. A. Hauser and D. S. LeBeau, "The Surface Structure and Composition of Colloidal Siliceous Matter," *Journal Physical and Colloidal Chemistry*, Vol. 55, p. 68–79. (1950).

(30) L. Michaelis, "Semiquinones, The Intermediate Steps of Reversible Organic Oxidation-Reduction," *Chemical Reviews*, Vol. 16, p. 243–286 (1935).

(31) J. B. Page, "Unreliability of the Benzidine Color Reaction as a Test for Montmorillonite," *Soil Science*, Vol. 51, p. 133–140 (1941).

(32) J. Endell, R. Zorn, and U. Hofmann, "The Benzidine Test for Montmorillonite," *Angewandte Chemie*, Vol. 54, p. 376–378 (1941). *Chemical Abstracts*, Vol. 35, p. 7878 (1941).

(33) D. Kruger and F. Oberlies, "Structure and Color Reactions of Montmorillonite Earths," *Naturwissenschaften*, Vol. 31, p. 92 (1943). *Chemical Abstracts*, Vol. 37, p. 4033 (1943).

(34) W. Siegl, "Detection of Montmorillonite with Benzidine," *Neues Jahrbuch für Mineralogie, Geologie, und Paleontologie, Monatsh*

A, Vol. 48, p. 40–43 (1945). *Chemical Abstracts*, Vol. 43, p. 1287, (1946).

(35) A. Esme, "Chemical Control of Bentonites by Color Test for Montmorillonite," *Annales Chimie Analytique*, Vol. 28, p. 31 (1946). *Chemical Abstracts*, Vol. 40, p. 2762.

(36) R. C. Mielenz, M. E. King, and N. C. Schieltz, "Staining Tests on the Reference Clay Minerals," Am. Petroleum Inst., Project 49, *Preliminary Report No. 7*, p. 135–160 (1950).

(37) H. E. Merwin and E. Posnjak, "Clays and Other Minerals from the Deep Sea, Hot Springs, and Weathered Rocks," *American Journal of Science*, 5th Series, Vol. 35A, p. 179–184 (1938).

(38) C. W. Correns and M. Mehmel, "Uber den Optischen und Rontgenographischen Nachweis von Kaolinit, Halloysit, und Montmorillonit," *Zeitschrift für Krist.*, Vol. 94, p. 337 (1936).

(39) American Petroleum Institute, New York, N. Y., Project 49, Clay Mineral Standards, *Preliminary Reports Nos. 1 through 8* (1949–1950).

(40) E. M. Chamot and C. W. Mason, "*Handbook of Chemical Microscopy*," John Wiley and Sons, Inc., New York, N. Y., Vol. 1, 2nd Ed., 478 p. (1938).

(41) M. S. Main, "Microscopic Examination of Reference Clay Mineral Specimens," Am. Petroleum Inst., Project 49, *Preliminary Report No. 5*, p. 15–58 (1950).

(42) G. F. Walker, "Vermiculite-Organic Complexes," *Nature*, Vol. 166, p. 695–696 (1950).

(43) C. D. Jeffries and M. L. Jackson, "Mineralogical Analysis of Soils," *Soil Science*, Vol. 68, p. 57–73 (1949).

(44) H. G. Dion, "Iron Oxide Removal from Clays and Its Influence on Base-Exchange Properties and X-ray Diffraction Patterns of the Clays,"*Soil Science*, Vol. 58, p. 411–424 (1944).

(45) C. J. Leith, "Removal of Iron Oxide Coatings from Mineral Grains," *Journal Sedimentary Petrology*, Vol. 20, p. 174–176 (1950).

(46) W. M. Clark, "*The Determination of Hydrogen Ions*," Third Ed., The Williams & Wilkins Co., 717 p. (1928).

PHYSICAL PROPERTIES OF PLASTICIZED SULFUR CEMENTS*

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Synopsis

In tests to determine the physical properties of plasticized sulfur cements, the variables studied were pouring temperature, reheating and aging effect, creep, and stress-strain relationships in tension and compression.

The test results indicate that there is an optimum pouring temperature, that the material may be reheated several times without changing properties, but that continued heating changes its properties, that there is an aging effect, that the stress-strain data produce a curve similar to that of normal portland-cement concrete, and that although creep is evident, almost full recovery can be expected with a sufficient time lapse.

In July, 1949, the Atlas Mineral Products Co. initiated a research program to determine the physical properties of plasticized sulfur cements. Some of the aims of this research project were to establish test techniques for corrosion, tension, compression, and torsion so that a typical sulfur cement pipe joint could be subjected to field conditions and the data derived therefrom analyzed. With this aim in mind, a basic testing program was established so that a correct interpretation of final results could be made. Although the program will eventually include many types of sulfur cements, all the tests described herein were performed on a particular proprietary sulfur cement plasticized with 1.2 per cent Thiokol by weight. The intent was to confine this portion of the program to one material until pouring, casting, and testing techniques could be established and verified.

There were five series of compression tests performed:

1. A series to determine the effect of pouring temperature on the ultimate compressive strength and ductility.
2. A series to determine whether the broken specimens of the above series would have the same stress-strain characteristics if remelt and recast in the same manner and temperature as the above series.
3. A series to determine the repetitive continuous heating effect on stress-strain characteristics.
4. A series to determine any aging strength effect after specimens had been cast.
5. A series to determine creep-time effects under various loads.

From the results of data on the compression tests, a tension test series was conducted at a particular pouring temperature, age, and rate of loading to determine the sulfur cements' stress-strain qualities in tension.

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All specimens subjected to test as reported in this paper were taken from a single sampling from the manufacturer's product.

Although shear, torsion, strength as a capping material for test cylinders, corrosion, and long-time pipe joint studies are an integral part of the program, only the studies on the tension and compression tests are completed and included herein.

The information published or submitted to appropriate journals for possible publication as a result of this research program include a brief history and a comparative report on various jointing materials and methods (5)[5]; directions for the use of sulfur compounds for jointing bell and spigot cast iron pipe (6); an investigation of the effects of capping materials on the apparent strength of concrete test specimens (1); the development of torsion testing equipment for cylindrical specimens (2); corrosion studies of iron in the presence of sulfur (3); and recent research in the performance of sulfur jointing compounds (4).

METHODS OF CASTING SPECIMENS

Several different procedures for casting the compression coupons were attempted before a split cylindrical steel mold was adopted. Specimens cast in standard 2-in cube molds gave inconsistent results when tested in compression. Examination after fracture showed voids to exist in this type of pour. These voids were probably caused by the shrinkage that occurs when sulfur cements change from the liquid to the solid state. Tests verified the opinion that compression coupons should be cut from a casting made by the use of a long square or cylindrical mold. Pyrex tubing with an inside diameter of $1\frac{1}{2}$ in., a length of 24 in., and with a greased inner surface was used as a mold, but this procedure was abandoned because slight irregularities in the tube made removal difficult. A rectangular prism type of mold was not used because machining

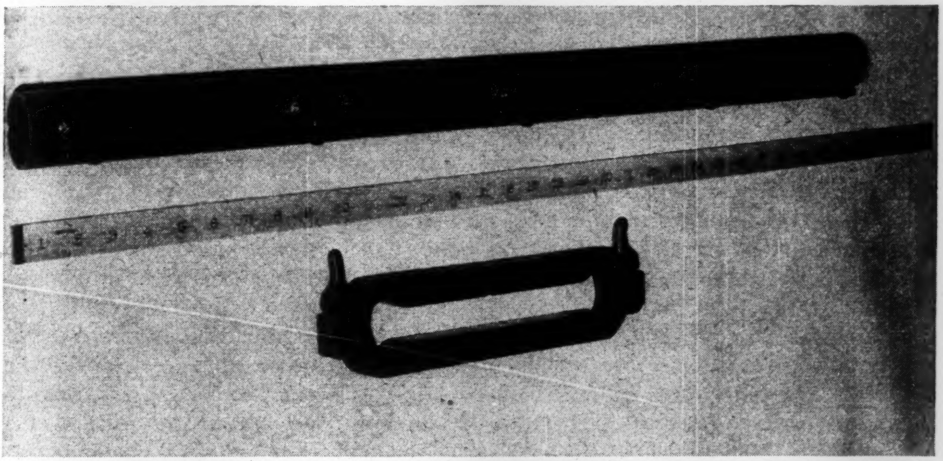

FIG. 1.—*Top*—Mold for Casting Compression Coupons. *Bottom*—Mold for Casting Elongated Tension Test Briquets.

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[5] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1247.

would have been expensive and troublesome.

A split cylindrical steel mold 30-in. long with an outer diameter of 2 in. and a wall thickness of $\frac{1}{8}$ in. proved to be a very convenient and satisfactory form for casting compression coupons. The mold shown in Fig. 1 was fabricated by milling a slot in 2-in. tubing that is manufactured to very close tolerances and is commercially available. Such a mold will yield as many as six 2 by 4-in. cylinders. Thus, a sufficient quantity of specimens may be obtained for any one test from one casting. Immediately prior to the pouring of the molten sulfur compound, the inner surface was greased with a silicone emulsion. Then lugs, which had been welded to the tube, were tightened sufficiently to close the milled slot. Then the cylindrical mold was positioned so that its axis was vertical and filled to the top with the material to be tested. As the sulfur compound cooled, a large shrinkage cone developed at the top, but the remainder of the specimen was free of voids. The reduction in volume occurring with the change from the liquid to solid state may be easily obtained by finding the volume of this shrinkage cone. Tests conducted in this manner showed the final shrinkage to be increased by 2 per cent for every 20 C rise in pouring temperature. After the mold had cooled, the solid cylindrical specimen was removed by simply loosening the mold lugs and sliding the cylinder out of the form. From these castings, specimens were cut to a length of 4 in. by the use of a carborundum wheel. All specimens were capped with a 50:50 mixture of high-early-strength portland cement and plaster of Paris to assure even load distribution. A capping jig was especially fabricated for this operation. Although not attempted, it is conceivable that an even better cap could be obtained by simply capping the coupon with molten sulfur cement.

The tension coupons for stress-strain measurements were cast in the elongated briquet-mold appearing in Fig. 1. This mold was fabricated by milling out the separators in a standard three-coupon briquet mold. The inner walls were machined so that a $\frac{1}{4}$ by 1-in. brass filler plate could be inserted on each side.

These plates were paralleled by means of set screws. A true open cross-section, 1 by 1-in., resulted with sufficient length to permit the mounting of either mechanical or bonded wire resistance strain gages on the casting without the danger of stress concentrations producing false strain indications.

Immediately prior to pouring, the elongated tension test specimen mold was greased and placed on a greased ground plate. A smooth $\frac{1}{8}$-in. plate was placed over the top rectangular portion of the mold. The coupon was cast by pouring molten sulfur cement into one of the open ends of the mold until the liquid had filled the form and risen to the top of the other open end. After the elapse of several seconds, a shrinkage hole began to appear at both of the open ends. Into the hole at one end more of the molten material was poured until the hole at the other end was filled. This process was repeated until shrinkage was no longer visible. A sound elongated tension briquet was thus obtained. Coupons showing any evidence of irregularities in the 1-in. square cross-section were discarded.

TESTING PROCEDURE AND RESULTS FOR COMPRESSION STUDIES

Pouring Temperature Effect:

This test series was conducted to determine the effect of pouring temperature on ultimate compressive strength and ductility as well as stress-strain characteristics. A batch of sulfur cement was cast in cylindrical glass molds at temperatures of 110, 130, 150, 170, and 190 C. The glass mold had a diameter of

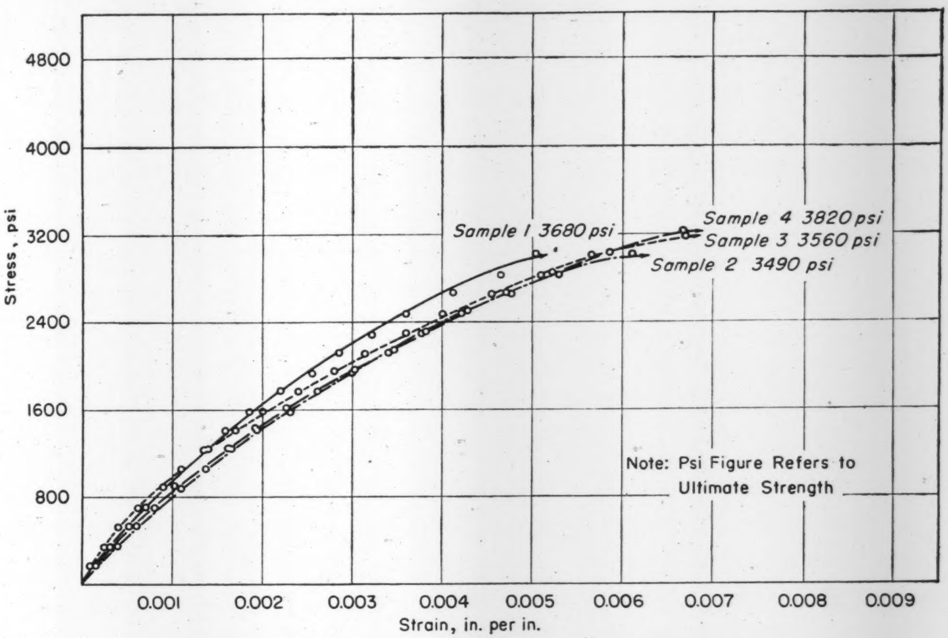

FIG. 2.—Stress-Strain Curves for a Pouring Temperature of 130 C.

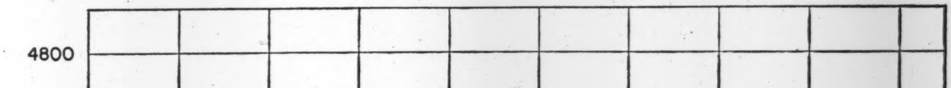

FIG. 3.—Stress-Strain Curves for Various Pouring Temperatures.

approximately $1\frac{1}{2}$ in., was approximately 24 in. in length, and was positioned for pouring so that its axis was vertical. From these castings, specimens were cut and capped as previously described. At least four specimens per each pouring temperature were tested in compression in a 300,000-lb Baldwin-Southwark hydraulic testing machine located at the Fritz Engineering Laboratory of Lehigh University at a continuous rate of loading of 1600 lb per min. This time interval was chosen merely to facilitate dial readings. Strains were observed over a central 2-in. gage length with a dial indicator attached to a Moore extensometer.

Stress-strain curves for each specimen were plotted as illustrated on Figs. 2 and 3. To meet publication requirements, only sample curves have been included. Figure 2 is a typical set of curves for four specimens poured at a temperature of 130 C. These figures reveal: (1) all specimens poured at one temperature form a definite band of curves, illustrating the consistency of test results, and (2) at only a pouring temperature of 170 C is stress proportional to strain; all other compression tests have a curved stress-strain plot. Figure 3 was drawn by replacing each set of curves for a single pouring temperature with an average curve. Thus a composite set of curves was drawn to study pouring temperature effects. The arrows indicate that the strain gage was removed at this point and load was continued to failure.

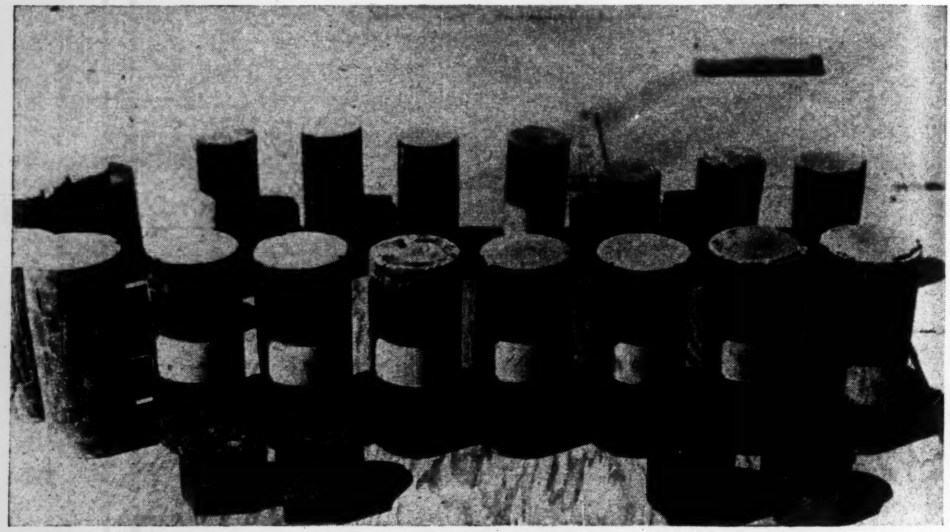

FIG. 4.—Typical Compression Coupon Failures.

Figure 3 reveals for the strains measured that (1) generally as the pouring temperature increases from 110 to 170 C the jointing compound loses strength, (2) for any given load the stiffness of the material generally decreases as the pouring temperature increases in the 110 to 170 C range, and (3) at a pouring temperature of 190 C the material suddenly gains in stiffness over the 170 C pour and at the same time increases greatly in strength over specimens poured at 170 C. Typical compression coupon failures are shown in

Figure 4. A double cone failure is characteristic of the material tested.

The most suitable pouring temperature for Thiokol plasticized sulfur cement appears to be in the neighborhood of 130 to 150 C because at that temperature it exhibits its best combination of strength, stiffness, and ductility.

Upon failure, all specimens emitted a rather offensive odor. The question arose as to whether the chemical properties of the material had changed under compression. To answer this question a second series of tests was run on these cylinders as described below.

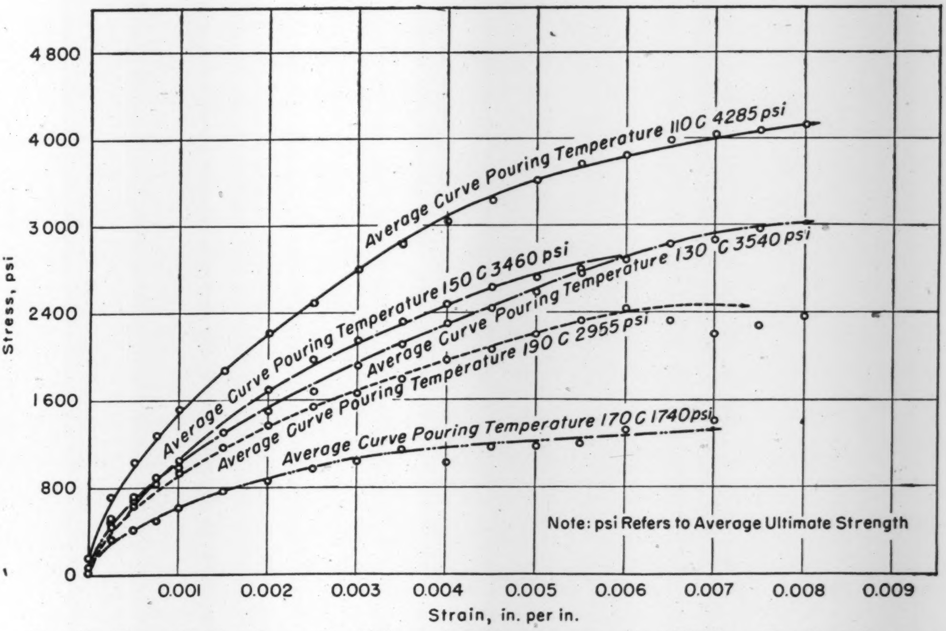

FIG. 5.—Stress-Strain Curves for Reheated Sulfur Compounds at Various Pouring Temperatures.

Remelting Effect:

This series was conducted to determine whether the broken specimens of the pouring temperature effect series would have the same stress-strain characteristics if remelted and recast in the same manner and at their previous temperature. The specimens of this series were allowed to age two to three days after casting as had those of the former series. After testing in compression, the stress-strain results were plotted for each pouring temperature and a final set of curves, Fig. 5, were drawn by replacing each set of curves for a particular pouring temperature by an average curve. If these figures are compared with the corresponding curves (Fig. 3), one observes: (1) for pouring temperatures of 110, 130, and 150 C, the latter series compares very closely with the former series, (2) the 170 C stress-strain curve is no longer a straight line and its ultimate strength has decreased appreciably with reheating, (3) reheating the 190 C specimens resulted in greater ductility and less strength, and (4) for the 130 C pouring temperature, the material did not appreciably change its stress-strain properties. It can be concluded that for a pouring temperature of 130 C the compressive characteristics are not changed due to the emission of the offensive odor discovered in the former series or by reheating. The same conclusion could be drawn for pouring temperatures of 110 C and 150 C but not at higher temperatures.

For this series a split cylindrical steel mold was used instead of Pyrex tubing which had been used in all previous series of tests. The mold had a $1\frac{3}{4}$-in. inside diameter and was 30 in. long. All cylinders were cast with the axis of the mold in a vertical position. They were cut and capped as previously described.

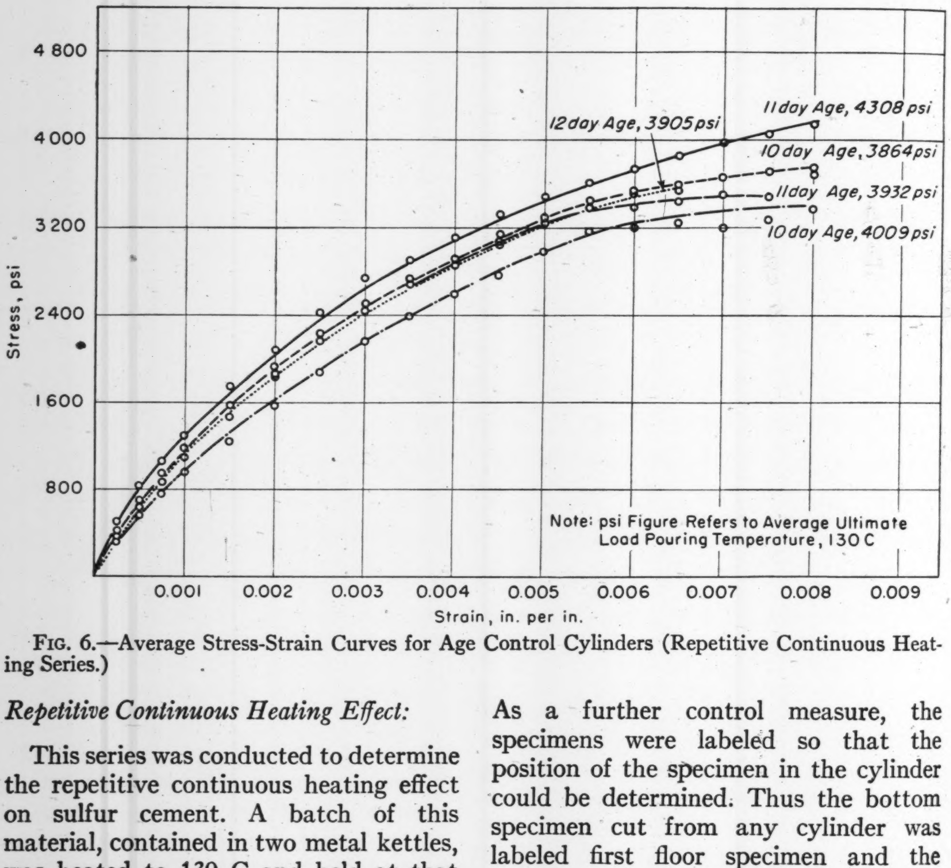

FIG. 6.—Average Stress-Strain Curves for Age Control Cylinders (Repetitive Continuous Heating Series.)

Repetitive Continuous Heating Effect:

This series was conducted to determine the repetitive continuous heating effect on sulfur cement. A batch of this material, contained in two metal kettles, was heated to 130 C and held at that temperature for 8 hr. At the end of this time a cylinder, labeled first cycle, was cast at 130 C and the batch was allowed to cool for 16 hr. The above procedure was repeated four more times, that is heated to 130 C and one cylinder poured at the end of each reheating cycle at 130 C. The cylinders were labeled consecutively, second through fifth cycle. As a further control measure, the specimens were labeled so that the position of the specimen in the cylinder could be determined. Thus the bottom specimen cut from any cylinder was labeled first floor specimen and the number of the specimen progressed consecutively as its relative position in height of pour increased.

At least four coupons per each cycle were tested to failure and stress-strain data were recorded by the use of a Moore extensometer with a 2-in. gage length.

Since the coupons of this series would have cured for various time intervals on the testing date, another series of control castings was poured concurrently so that aging effects could be interpreted. A freshly melted quantity of sulfur cement, which had not been subjected previously to heating or melting was cast and tested each time a pour or test was made from the repetitive continuous heating series. Again the position in the mold of the aging control coupons was noted. The repetitive continuous heating series and aging control coupons were tested to failure at an age of from ten to twelve days. Average stress-strain curves (Fig. 6) resulting from the control coupon data showed a negligible time-of-age variation effect. Therefore, it was concluded that the repetitive continuous heating average stress-strain curves (Fig. 7) needed no adjustment because of a variance in age of specimen at time of test.

The individual specimen curves for each heating cycle as well as those obtained from the control cylinder castings showed that the position of the specimen in the mold had no observable effect on either ultimate strength or stiffness. In order to conserve space, only the average curves of the repetitive continuous heating series have been included as shown in Fig. 7.

A study of Fig. 7 reveals:

1. The repetitive continuous heating effect is scattered and difficult to interpret. By comparing Fig. 7 with Fig. 6, it is apparent that continuous heat at 130 C for 8 hr causes the sulfur compound to lose strength and stiffness per any given applied load. For some reason not easily explained, the material seemed to recover its former properties when it was allowed to cool for 16 hr and then reheated for 8 hr. This effect is noticeable by comparing the first and second cycle curves of Fig. 7 with those of Fig. 6. The second cycle curve is in general agreement with those of Fig. 6,

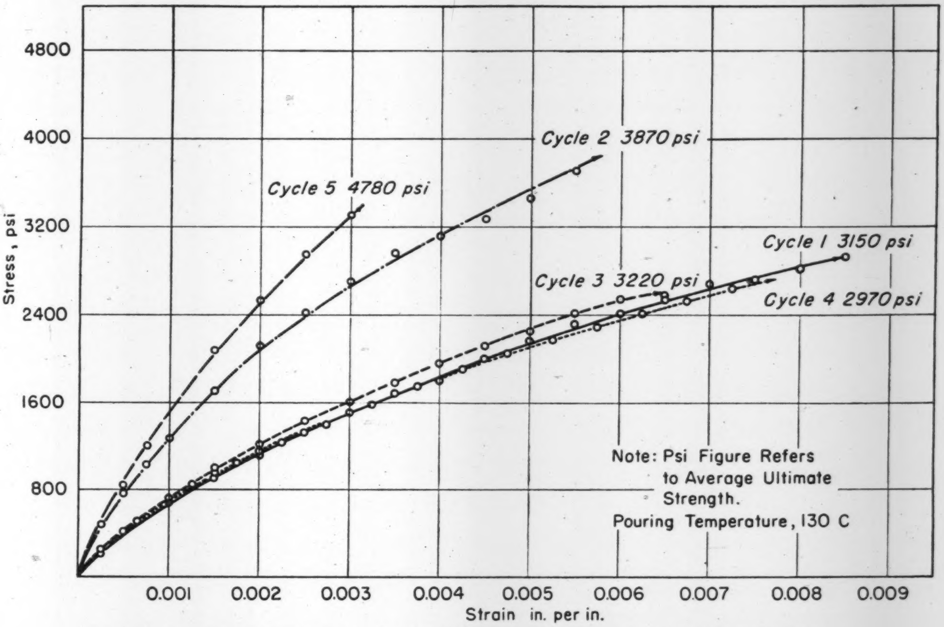

FIG. 7.—Stress-Strain Curves for Repetitive Continuous Heating Cycles.

indicating that the material has the same stress-strain pattern and ultimate strength as new material exhibits. Specimens cast at the end of the third and fourth heating cycles have about the same characteristics as those cast after the first heating cycle. Specimens cast after the fifth heating cycle increase greatly in ultimate strength and stiffness.

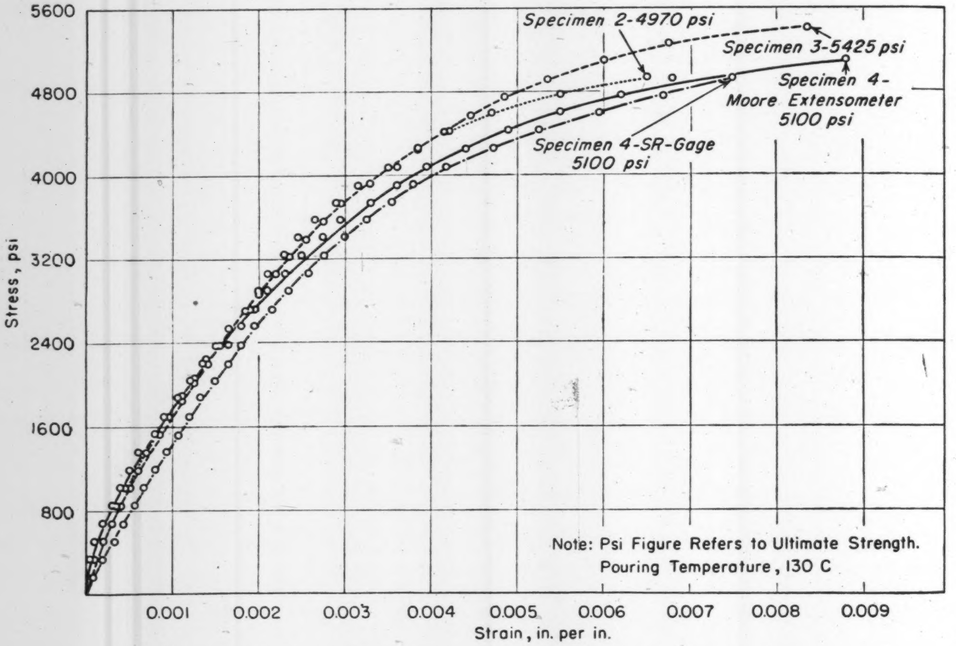

FIG. 8.—Stress-Strain Curves for 30-Day Old Cylinders.

2. It seems safe to say that for the best stiffness consistent with reasonable strength, Thiokol plasticized sulfur cement should be heated to 130 to 150 C pouring temperature and then poured as soon thereafter as possible. Just how long the heat can be maintained without adverse effects is not known; holding at 130 C for 8 hr caused the physical properties to change. Certainly old material which has remained in the melting pot under continuous heat for an extended time should be discarded if the original physical properties are desired.

Aging Effect:

A series of tests was performed to determine the effect of aging on stress-strain curves. At least three specimens in each age group were cast, capped, and tested as previously described. After the cylinders were cast in a split mold at a pouring temperature of 130 C, they were cured at room temperature and humidity for periods of 1, 2, 3, 4, 9, 11, 30, 60, and 90 days respectively before testing to failure in compression.

In the previous tests, the Moore extensometer, which is a mechanical strain gage, was removed when failure appeared imminent so that it would not be damaged by a sudden failure. Thus, it usually was not possible to record strains near the breaking load. So that complete stress-strain curves from load inception to failure could be recorded, bonded wire resistance strain gages as well as the Moore extensometer were placed on some of the aging effect series of coupons. Type A-7, SR-4 strain gages were used and strains were recorded by use of a Type K, Baldwin-Southwark portable SR-4 strain indicator. When possible, two SR-4 gages were cemented with cellulose acetate cement to at least one specimen from each age group. These gages were centered at 180 deg apart circumferentially and arranged so that the recorded strain was parallel to the line of load application. By using a series connection of lead wires, only one strain reading was necessary for each increment of load. In addition, the Moore extensometer was mounted at 90 deg circumferentially to the SR-4 gages. This provided a mechanical strain gage and SR-4 strain gage check for sulfur cements.

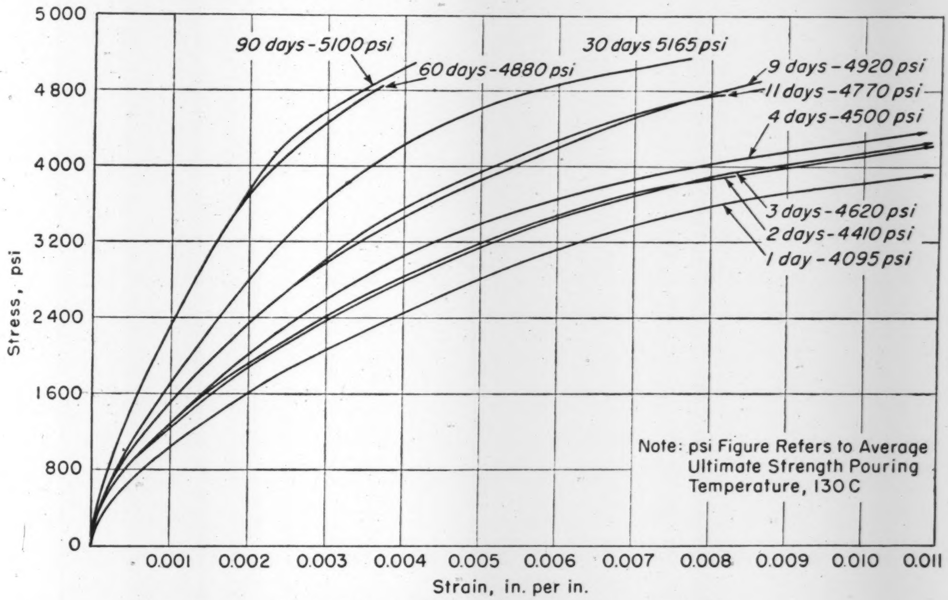

FIG. 9.—Average Stress-Strain Curves Showing Aging Effect.

Figure 8, a 30-day age effect test, illustrates a typical group of stress-strain curves obtained as described above on three specimens. No strains were recorded on specimen No. 1 because it was used to indicate only the ultimate compression at a 30-day age. One notes the close agreement between the mechanical gage and SR-4 gages which were mounted on specimen No. 4 of this particular age group. It is concluded that both the Moore extensometer and SR-4 strain gages are satisfactory for strain measurement on sulfur compounds.

The results of the aging test series are shown by the average stress-strain curves for various ages appearing on Fig. 9. It is apparent that strength and stiffness increase with age of material. Thus, it is essential that a standard age of test cylinder be adopted if comparable results are expected.

Creep Effect Series:

A series of compression creep tests was conducted to establish a proper rate of loading and to determine the rate of flow of compression specimens under sustained load. All of the specimens used in this test series were poured from a single batch of sulfur cement at a pouring temperature of 130 C. Specimens were cast, cut, and capped as previously described. Longitudinal strains were measured by two SR-4 strain gages mounted on a central circumference and separated by 180 deg. Possible bending strains were eliminated by connecting the gage leads in series.

The stress level, at which creep under sustained load was measured, was reached by applying load at a rate of 1600 lb per min. During this loading period the rate of loading was not interrupted. Continuous strain-load readings were recorded simultaneously as the applied force approached the desired stress level. After the stress level was reached, the load was maintained constant and held so for 1 hr. Strains were recorded throughout this period. At the end of 1 hr the specimen was unloaded, and strains, indicating the rate of recovery, were observed for a period of 1 hr.

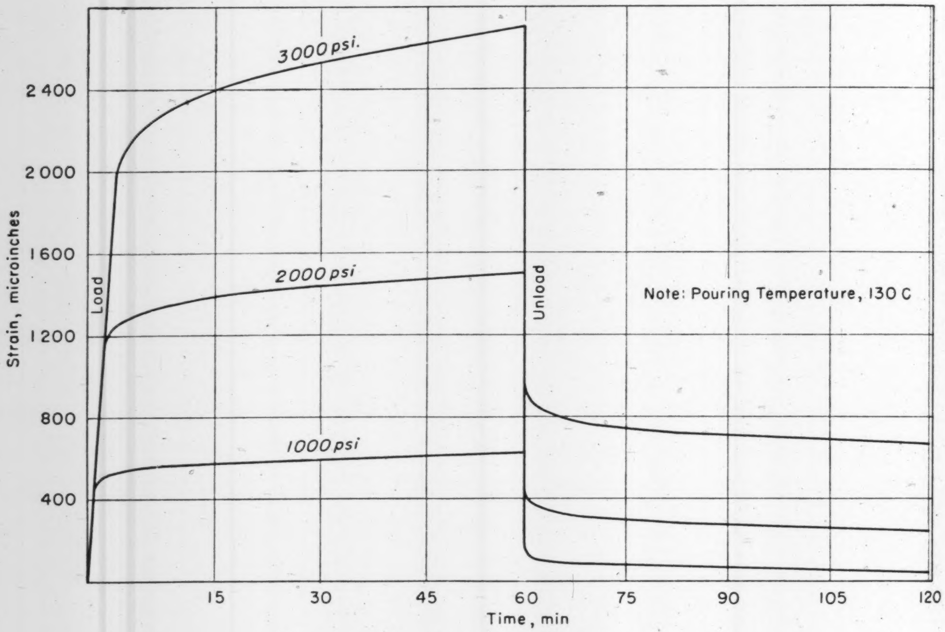

FIG. 10.—Creep Test Curves.

Tests were conducted at room temperature on a Dillon tension tester which was equipped with a compression cage and a dynamometer of sufficient range to accommodate the respective stress level. Compression creep tests were performed at seven different stress levels, namely, 500, 1000, 1500, 2000, 2500, 3000, and 3500 psi.

Figure 10 contains the creep test results for stress levels of 1000, 2000, and 3000 psi. These tests have revealed some interesting results. Stress-strain curves for sulfur compound tests can be greatly influenced by the rate of loading due to the material exhibiting a considerable creep under load. For stress levels up to 2000 psi, the strain increased roughly 25 per cent during a 1-hr period. For stress levels between 2500 and 3000 psi, the strain increased roughly 30 per cent during a 1 hr period. Although not shown on Fig. 10, the 3500-psi stress level yielded a 40 per cent increase in strain. All strain-time curves are of the same type. The initial rate of increase of strain with respect to time is large and decreases with time. After the load has been released, a residual strain exists. For specimens subjected to stresses up to 2000 psi, the residual strain is roughly of the same order of magnitude as the creep strains. For applied stresses over 2000 psi, the residual strains are slightly higher than the creep strains. After the initial strain recovery, the material continues to recover at a rate of from 16 to 13 per cent (based on the initial strain at zero time), for the first hour for applied stresses of 500 to 3500 psi respectively. The recovery in the second hour will probably be slower because the slope of the recovery curve decreases.

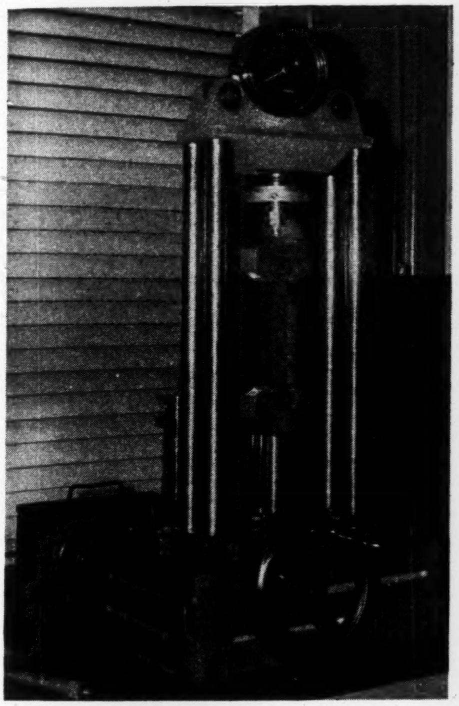

FIG. 11.—Elongated Tension Test Briquet in Dillon Tensile Tester.

The ability of the material to flow under stress is a very useful property in many applications.

TESTING PROCEDURE AND RESULTS FOR TENSION STUDIES

A series of elongated briquet tension specimens was tested to determine the stress-strain properties of the sulfur cement in tension. These coupons were cast in a manner previously described at a pouring temperature of 130 C. Figure 11 is a view of a specimen under tension in the Dillon tension tester. All specimens were loaded continuously at the rate of 120 lb per min. This loading rate was established through an endeavor to load at a rate proportional to the compression rate of loading based on the respective anticipated ultimate strengths in tension and compression. Strains were recorded by both Type A-7 SR-4 gages and the Moore extensometer. When SR-4 gages were used, they were mounted on the central portion of the rectangular part of the briquets so that they were on opposite faces. The gages were then wired in series to eliminate any bending stresses.

The results for one series of tension tests appear on Fig. 12. Stress-strain curves are of the same general shape as those of the compression series. It is recommended that only SR-4 gages be used on tension tests. The Moore extensometer tends to cause bending stresses due to its weight effect on the 1-in. square cross-section. In addition, the bonded wire resistance gage is capable of recording much smaller strains than the mechanical gage used and is desirable if the small ultimate stress in tension is to yield readable strains.

Tests on normal tension sulfur cement briquets conducted in a testing machine conforming to that described in ASTM Method C 109[6] show ultimate strengths of approximately 650 psi which is considerably higher than the values indicated on Fig. 12. Tension tests with two different rates of loading demonstrated that failure in tension at a low rate of loading may occur at approximately one-half the tensile strength usually obtained in the control laboratory where the rate of loading is 600 lb per min. Therefore, the rate of loading in a tension test is a critical item.

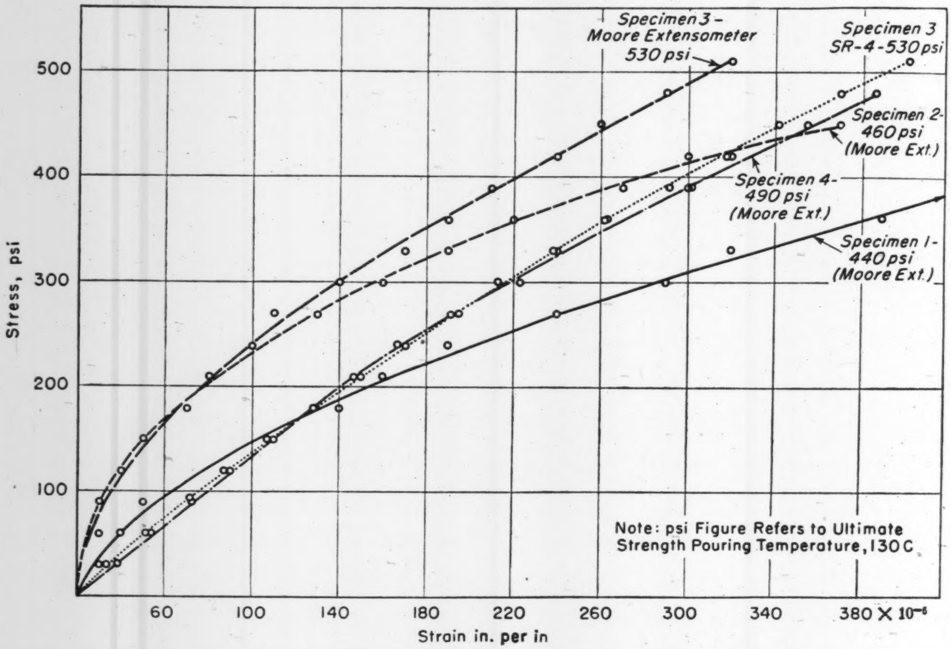

FIG. 12.—Tension Stress-Strain Curves for 9-Day Old Specimens.

CONCLUSIONS

The results of the tests performed on Thiokol plasticized sulfur cements lead to the following conclusions:

1. Sound compression coupons can be obtained easily by the use of a split cylindrical steel mold.

2. The most suitable pouring temperature appears to be in the neighborhood of 130 to 150 C because at that temperature the sulfur cement possesses the best combination of strength, stiffness, and ductility.

3. For a pouring temperature of 110 to 150 C the compressive characteristics are not changed by reheating and recasting at the previous pouring temperature.

4. It is essential that a standard age of test cylinder be adopted if comparable results are to be expected because both

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[6] Standard Method of Test for Compressive Strength of Hydraulic-Cement Mortars (C 109 – 50), 1950 Supplement to Book of ASTM Standards, Part 3, p. 26.

strength and stiffness of coupons increase with age.

5. Stress-strain curves and ultimate strengths for sulfur cements can be greatly influenced by the rate of loading because the material exhibits a considerable creep under sustained load. Therefore, for comparable results the rate of loading in compression or tension must be standardized.

Acknowledgment:

The authors wish to express their appreciation to the Atlas Mineral Products Co. for supporting and approving publication of the results of this investigation. The contributions of George L. Wirtz, Sr., Arthur L. Moser, Robert H. Steiner, and Robert D. Stout, are also gratefully acknowledged.

References

(1) F. M. Master, Jr., and A. C. Loewer, Jr., "An Investigation of the Effects of Capping Materials on the Apparent Strength of Concrete Test Specimens," Fritz Engineering Laboratory Library, Lehigh University.

(2) E. K. Muhlhausen, A. C. Loewer, Jr., W. J. Eney, "Torsion Testing Equipment for Cylindrical Cement Specimens," Fritz Engineering Laboratory Library, Lehigh University.

(3) R. B. Seymour, W. Pascoe, R. D. Stout, "Corrosion Studies of Iron in the Presence of Sulfur," *Corrosion*, Vol. 7, No. 8, Aug., 1951, pp. 265–268.

(4) R. B. Seymour, W. Pascoe, W. J. Eney, A. C. Loewer, Jr., R. H. Steiner and R. D. Stout, "Performance Studies on Sulfur Jointing Compounds" *Journal*, Am. Water Works Assn., Vol. 43, No. 12, Dec., 1951, pp. 1001–1014.

(5) R. B. Seymour, "Jointing Compounds for Sewer Pipe, "*Public Works*, Vol. 82, No. 8, Aug., 1951, p. 74.

(6) A. M. Younger and R. B. Seymour, "Recommended Practices in the Installation of Sulfur Compound Water Main Jointings," H_2O, Fall Issue (1949).

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON ACOUSTICAL MATERIALS

The papers in this Symposium discuss briefly the origin and history of ASTM Committee C-20 and outline the many problems which it is attempting to solve. The Symposium was held at the Twenty-fourth session of the Fifty-fourth Annual Meeting in Atlantic City, N. J., June 22, 1951.

The Symposium included the following papers:

Brief History of the Acoustical Materials Industry—Wallace Waterfall
Activity of Committee C-20 on Acoustical Materials—H. A. Leedy
The Measurement of Sound Absorption—Hale J. Sabine
Combustibility of Acoustical Materials—Wallace Waterfall
Maintenance of Acoustical Materials—L. F. Yerges
Basic Physical Properties of Acoustical Materials—William Jack

These papers together with discussion have been issued by the ASTM as Special Technical Publication No. 123 entitled "Symposium on Acoustical Materials."

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON CONSOLIDATION TESTING OF SOILS

This Symposium brought together the varied experiences in consolidation testing of soils and knowledge of the nature of consolidation phenonemona under varied controlling conditions in natural situations and as modified by structures and by construction methods and practices. It was presented at the Second Session of the Fifty-fourth Annual Meeting of the American Society for Testing Materials held in Atlantic City, N. J., June 18, 1951.

The following papers were presented:

Report of Consolidation Tests with Peat—L. A. Palmer and J. B. Thompson
Consolidation and Related Properties of Loess Soils—W. G. Holtz and H. J. Gibbs
Settlement of the Railroad Embankment Crossing of the Morganza Floodway, Louisiana—W. G. Shockley and C. I. Mansur
Aids in the Interpretation of the Consolidation Test—L. H. Matlock and R. F. Dawson
A Rapid Technique of Consolidation Testing—R. H. Karol
The Effect of Temperature on the Consolidation Characteristics of Remolded Clay—F. N. Finn
Observed Settlement Due to Consolidation of Alluvial Clay—E. S. Barber
The Application of Controlled Test Methods in Consolidation Testing—D. M. Burmister

These papers together with discussion have been issued by the ASTM as Special Technical Publication No. 126 entitled, "Symposium on Consolidation Testing of Soils."

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON SURFACE AND SUBSURFACE RECONNAISSANCE

Committee D-18 on Soils for Engineering Purposes sponsored this Symposium at the Tenth and Eleventh Sessions of the Fifty-fourth Annual Meeting of the American Society for Testing Materials held in Atlantic City, N. J., June 19, 1951. The symposium assembled information on techniques that have been applied to this type of reconnaissance in widely varying scientific fields and examined them for their limitations as well as applications.

The Symposium was composed of the following:

Interpreting Geologic Maps for Engineers—E. B. Eckel
Engineering Implications of Geological Reconnaissance in the Plains Area—Missouri River Basin—E. A. Abdun-Nur and J. D. Dowling
Preliminary Foundation Exploration in Arctic Regions—L. A. Nees and A. M. Johnson
Preparation of County Engineering Soil Maps for Illinois—T. H. Thornburn
The Engineer Looks at Pedology—R. L. Greenman
Application of Aerial Photographs to Preliminary Engineering Soil Surveys—R. D. Miles
The Preparation of an Engineering Soil Map of New Jersey—D. R. Lueder
Experience with Geophysics in New York State—P. H. Bird
Earth-Resistivity Tests Applied to Subsurface Reconnaissance Surveys—R. W. Moore
Electrical Resistivity Geophysical Method as Applied to Engineering Problems—H. L. Scharon
Geophysical Measurements of the Depth of Weathered Mantle Rock—D. Wantland
The Practical Value of an Earth Resistivity Method in Making Subsurface Explorations—W. F. Abercrombie
Application of Seismic Methods to Foundation Exploration—A. M. Johnson and R. H. Wesley
Seismology Applied to Shallow Zone Research—D. Linehan
Resistivity Reconnaissance—I. Roman

These papers together with discussion have been issued by the ASTM as Special Technical Publication No. 122 entitled, "Symposium on Surface and Subsurface Reconnaissance."

PROGRESSIVE HETEROGENEITY IN AGED "NEGATIVE SPOT-TEST" NAPHTHA SOLS*

By G. L. Oliensis[1]

Synopsis

In 1941, it was found that the more homogeneous the internal structure of an asphalt was, the longer would be its "negative period" (the time required for its spot-test sol to turn positive), and the latter value was reported for 18 of the 39 asphalts studied whose end point had by then been reached. During the ten years since elapsed, 15 of the remaining 21 asphalts have attained this end point.

This paper presents (*a*) a tabulation of all negative-period values obtained to date, (*b*) a 10-yr comparison between "negative period" and "hexane resistance" values, (*c*) a study of the comparative effectiveness of commercial hexane and pure normal hexane for the determination of "hexane resistance;" and (*d*) the bearing of the foregoing data on the character of the flocculated bodies.

In a paper read before this Society 10 yr ago,[2] the author described a series of spot tests on 39 different asphalts and asphalt blends from different geographical sources, the naphtha sols of which were "negative" to that test as ordinarily performed, but invariably turned "positive" after the addition of a sufficient number of increments of hexane, and in many cases also became positive merely on standing undisturbed for a sufficient time. As the number of hexane increments required for a given sol (that is, its "hexane resistance") increased, the time required for the same sol to turn positive spontaneously (that is, its "negative period") appeared also to increase as if in a roughly geometrical progression, each successive increment of hexane resistance corresponding to an ever larger increase in the length of the negative period. By the time the 1941 paper was released, only 18 of the 39 sols had reached the end of their negative period, in between 1 and 96 days; and these incidentally had also shown the lowest hexane resistance, namely, between 7 and 18 increments.

It was consequently decided to let the other 21 sols remain in their tightly-stoppered flasks in a dark laboratory compartment undisturbed except for periodic spot-test checks, at intervals which corresponded roughly to 10 per cent of the age of the sol—that is, at monthly intervals for approximately year-old sols, half-yearly intervals for those 5 yr old, and approximately yearly intervals for those 8 and 9 yr old. After each such interval the contents of the test flask were thoroughly stirred and a

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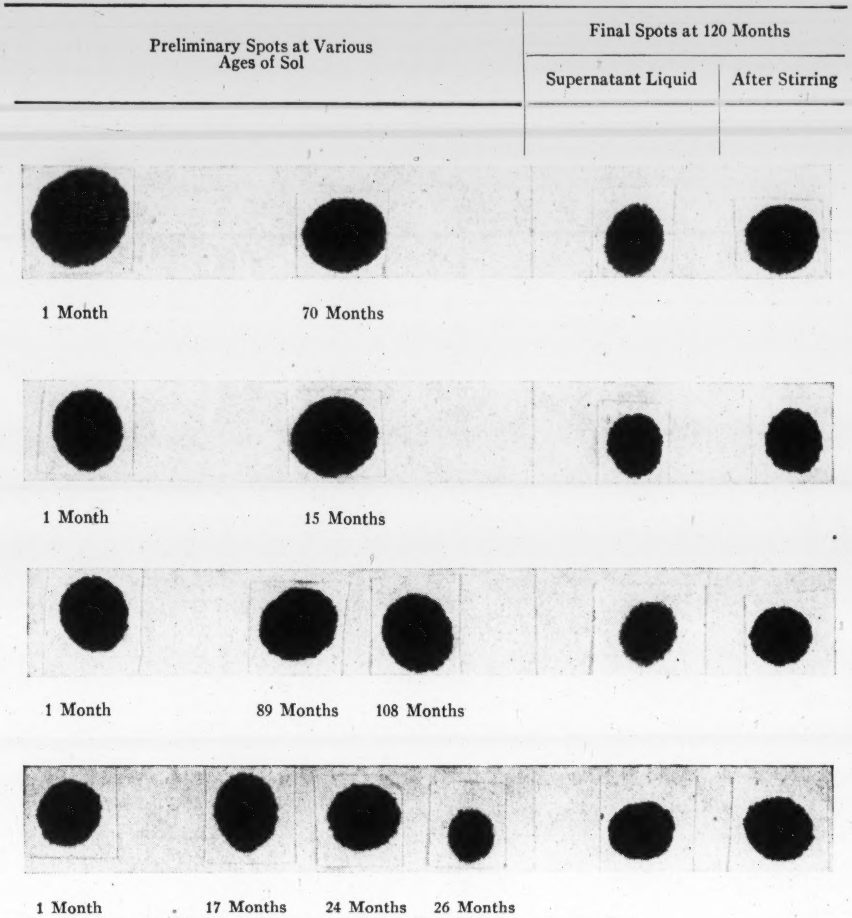
* Presented at the Fifty-fourth Annual Meeting of the Society, June 18-22, 1951.

[1] Director of Research, Lloyd A. Fry Roofing Co., Summit, Ill.

[2] G. L. Oliensis, "Fundamental Significance of Oliensis Spot Test—Quantitative Test for Homogeneity," *Proceedings*, Am. Soc. Testing Mats., Vol. 41, p. 1108 (1941).


Preliminary Spots at Various Ages of Sol
Final Spots at 120 Months
Supernatant Liquid
After Stirring
1 Month
70 Months
1 Month
15 Months
1 Month
89 Months
108 Months
1 Month
17 Months
24 Months
26 Months

(*a*) Ring-nucleus remains practically unchanged during last 4 yr (Item No. 18, Trinidad residual).

(*b*) Strong solid nucleus remains practically unchanged during last 9 yr (Item No. 2, Urbana residual, slightly overheated).

(*c*) Strong solid nucleus remains practically unchanged during 8th and 9th year; intensifies in 10th yr (Item No. 1, Smackover residual).

(*d*) Strong nucleus remains unchanged for half year (between 17 and 24 months); stabilizes as much blacker nucleus during last 8 yr (Item No. 30, Smackover residual plus 5 per cent cracking-coil tar No. 1).

Fig. 1.—Appearance of Spot at Different Stages of Negative Period of Some Typical Long-Term Sols.

drop was placed on a No. 50 Whatman filter paper, beside the test drops previously taken from the same flask. During the entire 10-yr period about 75 drops were so withdrawn—half of them in the first 3 or 4 months. Though no doubt some evaporation and aeration occurred each time a sol was stirred and sampled, concentration and thickening must have been relatively slight, as the shade of

TABLE I.—QUANTITATIVE HOMOGENEITY TESTS.

Item	Crude Oil Source	Refinery	Age of Sample at Start of Test	Penetration at 77 F	NEGATIVE PERIOD						HEXANE RESISTANCE
					Short-Term Sols, days	Long-Term Sols, Months					Increments
						Stage A (Traces)	Stage B (Very Faint)	Stage C (Faint but Definite)	Stage D (Strong)	Stage E (Very Strong)	
(*A*) STRAIGHT REDUCED RESIDUALS											
No. 1	Arkansas (Smackover)	A	Recent	45		12	23	59	89	120	30 or 31
	Arkansas (Urbana)										
No. 2	Sli. overheated	B	Recent	76		7	9	11	15–120		20
No. 3	Sli. overheated (?)	B	Recent	87		8	13	15	18–120		22
No. 4	Normal	B	Recent	88		14	45–120				36
No. 5	California	C	6 yr	Soft	7						9
No. 6		D	Recent	55		12–120					37
No. 7	Kansas	E	Recent	45		8	36–58	64–120			51
No. 8	Kansas	E	Recent	150		9	36–120				61
No. 9	Kansas	F	2 months	52		9	36	120			41
No. 10	Kansas	F	1 wk	50		15	46–120				43
No. 11	Kansas (blend)	F	1 month	43		7	8	12	120		30
No. 12	Mexican	G	14 yr	45	67						12
No. 13		G	14 yr	175		4	5	5.3	5.5	5.7–6.5	18
No. 14		H	9 yr	50	(89 to) 102						18
No. 15		H	4 yr	150		5	8	9.0	9.5	10–11	22
No. 16	Texas (Hendricks)	J	4 yr	45		5	12	19	120		29
No. 17		J	Recent	90		20	36–120				34
No. 18	Trinidad Oil Resid.	K	Recent	55		8	10	39	70–120		23
No. 19		K	Recent	155		6	9	39	70–120		27
No. 20	Venezuela	L	11 yr	30	(2 to) 3						7
No. 21		M	Recent	55	25						16
No. 22		M	Recent	155	25						16
No. 23		N	Recent	85		6	58–120				36
No. 24	Wyoming (overheated)	P	2 yr	325	(2 to) 3						7
No. 25	Wyoming (normal)	P	2 yr	208	(65 to) 72						14
No. 26	Wyoming (normal)	P	2 yr	37	96						16
No. 27	Wyoming	Q	Recent	45	34						14
No. 28		Q	Recent	155	31						14
No. 29	Synthetic	R	Recent	85		3	6	16–120			22

(*B*) LABORATORY BLENDS WITH CRACKING-COIL TAR

Item	Smackover (Item 1), per cent	Cracking Coil-Tar, per cent		Short-Term Sols, days	Stage A (Traces)	Stage B (Very Faint)	Stage C (Faint but Definite)	Stage D (Strong)	Stage E (Very Strong)	Hexane Resistance Increments
		No. 1	No. 2							
No. 30	95	5			6	9	13	17	26–120	23
No. 31	90	10		(73 to) 83						18
No. 32	80	20		3						7 or 8
No. 33	75	25		0 to 1						
No. 34	95		5		9	14	32	43	46–120	27 or 28
No. 35	90		10		5	9	12	16	24–120	24
No. 36	80		20	(49 to) 56						17
No. 37	70		30	9						...
No. 38	65		35	3						...
No. 39	60		40	0						0

clear brown left by the drop on the filter paper at the end of the 10-yr period was only in rare cases appreciably darker than that left by drops from the same sample in 1941 (compare the 1-month with the 120-month spot in each of the 4 rows in Fig. 1).

In the course of this investigation, it became evident that the longer the negative period ultimately proved to be, the vaguer were the initial indications of heterogeneity and the more difficult it was to determine each end point. It was therefore decided not to attempt to determine the latter exactly, but instead to record the age of the sol when it first reached each of the following successive stages: (*a*) when a ring or nucleus became visible as a vague trace that occasionally failed to recur in successive spots; (*b*) when the ring or spot began to recur regularly, though still very faintly; (*c*) when it first became definite enough to be unmistakable, even though still rather faint; (*d*) when it became fairly strong; and (*e*) when and if it increased to a markedly intenser black than in the stage preceding. The beginning of stage (*c*), when the spot is described as "faint but definite," is probably the closest approach possible (in the case of the long-term sols here being discussed) to the negative period as estimated for the shorter-term sols in the 1941 paper.

Table I contains the results obtained on the 39 samples during the entire 10-yr investigation. It is arranged in most respects like Table I of the 1941 paper, except for the necessity of subdividing the negative period into the several stages just mentioned. It will be noted that of the 21 sols still negative 10 yr ago, 15 more have now reached stage (*c*), which as explained corresponds roughly to the end of the negative period, and the remaining 6 have also developed either very faint or traces of nuclei, though the latter have not yet become definite enough to warrant classifying their sols as positive.

The final 10-yr results in Table I confirm in the main the conclusions reached in the 1941 paper, namely, that when asphalts from the same general source or refining process, or asphalt blends of the same general type, are compared with one another, it is noted that as the curve of the hexane resistance rises the curve of the negative period rises correspondingly, but very much more steeply, so that with hexane resistances of 30 or more increments, negative periods of 5 to 10 yr and more are quite frequent. Furthermore, the more carefully refined an asphalt is, or the freer from contamination with positive or cracked asphalts, the higher is the hexane resistance and the longer the negative period. Definite trends in that direction had already become apparent from the preliminary data in the 1941 paper, but the more extensive data now available enables one to follow these trends much farther.

Thus in the case of Items 24, 25, and 26, representing one overheated Wyoming residual and two normally refined residuals from the same source, there is noted a slowly increasing hexane resistance of 7, 14, and 16 increments and a corresponding but more rapidly lengthening negative period of 3, 72, and 96 days.

Again in Items 30 to 32, inclusive, representing a group of blends of a Smackover residual with from 5 to 20 per cent of a harshly treated cracking coil tar No. 1, it is noted that as the tar content diminishes the hexane resistance rises rapidly from 8 to 23, and the negative period rises still more rapidly from 3 days to 13 months.

Again in Items 34 to 38, inclusive, representing a similar group of Smackover blends with from 5 to 35 per cent of the somewhat milder cracking coil tar No. 2, it is noted that as the tar content diminishes, the hexane resistance rises from 7 to 28, and the negative period curves up steeply from 3 days to 32 months, though even the latter value, high as it seems to be, falls far short of the 59 months value shown by the straight Smackover residual.

The most striking illustration of all, however, is found in Items 2, 3, and 4, representing a group of Urbana residuals of 85 penetration, of which the first had been reported as having been somewhat overheated, the second had been suspected of having been very slightly overheated, while the third had been refined in the normal manner with no evidence of overheating whatever. It is noted that these three samples show a slowly increasing hexane resistance of 20, 22, and 36, while the negative period soars upward sharply from 11 to 15 months and to better than 120 months. Both the hexane resistance and the negative period determined on these three samples indicate pointedly that the second sample, which had been merely suspected of having been very slightly overheated, must have actually been overheated to nearly the same degree as the first. It is clear therefore that, as stated in the concluding paragraph of the author's 1941 paper, these two tests can detect and measure even slight deviations of an asphalt from its "norm," either when these are due to differences in severity of treatment during processing or to blending or contamination with asphalts of a different degree of homogeneity; hence they have great potential value both to the plant control chemist and to the researcher.

PURE AND COMMERCIAL HEXANE

The type of hexane used in all the hexane resistance tests so far described is a commercial grade known as Skellysolve "B" obtained from the Skelly Oil Co., Kansas City, Mo. There is a purer but much more expensive type obtainable from the Phillips Petroleum Co., Bartlesville, Okla. The physical and chemical characteristics of each are given in Table II, mostly from data recently submitted by the producers and released here with their permission, though the aniline number and the negative period and hexane resistance values were determined in the author's laboratory.

TABLE II.

	Skellysolve "B"	Phillips Pure Normal Hexane
Gravity A.P.I. at 60 F..	75.2	81.6
Flash point, closed cup.	−20 F	...
Reid vapor pressure, psi at 100 F..........	5.1	4.96
Aniline No.............	148.5 F	(Still turbid at 158 F)
Boiling point...........	...	155.8 F
ASTM Distillation:		
Initial boiling point..	146 F	...
50 per cent over....	149 F	...
95 per cent over....	153 F	...
Dry point..........	156 F	...
Chemical Composition, per cent:		
Normal hexane.......	40.9	99.30
3 Methylpentane.....	11.7	.49
2 Methylpentane	25.5	...
Methyl Cyclopentane.	17.3	.20
Benzene..............	1.4	.01
Others................	3.2	...
	100.0 per cent	100.00 per cent

Negative Period versus Hexane Resistance:

Source	Refinery	Penetration at 77 F	Negative Period, days	Hexane Resistance, Increments	
				Skellysolve "B"	Phillips Pure Normal Hexane
Calif. Steam-refined					
No. 1...........	S	74	4	(7 to) 8	(6 to) 7
No. 2...........	T	390	33	(16 to) 18	(13 to) 14
Mid-Continent steam-vacuum					
No. 1...........	U	85		18	14
No. 2...........	V	85	184+	(43 to) 44	30
Propane Residuum No. 1					
1st Test.........	W	90	150+		(39 to) 41
Check Test......	W	90	...		(38 to) 40
Propane Residuum No. 2					
1st Test.........	X	735	150+		(37 to) 40
Check Test......	X	735	...		(38 to) 40

It will be seen from Table II that the pure normal hexane is much higher in straight normal paraffins than is Skellysolve "B." This no doubt accounts for its higher aniline value. It is true that that value for the pure normal hexane cannot be determined exactly since it happens to be higher than the boiling point. However, since the blend of aniline and the normal hexane was found to be still turbid at a temperature of 158 F when it was boiling vigorously, it can be assumed that its aniline number was definitely higher than that temperature, and hence is at least 10 F higher than that of Skellysolve "B." It follows that the normal hexane should have a greater flocculating, or desolvating, power than the other, and it is therefore not surprising that the hexane resistance figure as determined with it is consistently lower than that obtainable with Skellysolve "B."

Since in the hexane resistance test the increments are added at 5-min intervals, considerable time can manifestly be saved when the pure normal hexane is used. Furthermore, its high purity and uniformity in composition make it possible to obtain very close checks in the resistance values determined with it, as is borne out by the tests on the two propane residuums at the foot of Table II. Each of them was tested twice, a month apart, with the operator unaware during the second set of tests of the results obtained previously; yet the hexane resistance values determined both times on each of the two propane residuums were almost identical.

For the foregoing reasons the pure normal hexane might be considered the more logical of the two reagents for rapid and accurate hexane resistance work, but its disadvantage lies in its extremely high price, which is $8.50 a qt Skellysolve "B," though not quite as pure, may therefore be selected by some laboratories as the more practical solvent for routine work, for though it does take longer to complete a test with it, especially when the sol happens to be highly resistant to flocculation, its price is moderate, and past work with a supply furnished by its producer has demonstrated that it too is capable of yielding satisfactorily concordant values—in every case somewhat higher than those obtained with the Phillips product.

BEHAVIOR OF ASPHALT-NAPHTHA SOLS DURING AGING

The first indication of heterogeneity in these sols is a trace of fine black sediment in the circular depression in the bottom of the flask, observable when the latter is tilted, and easily dispersed again with a few swirls of the liquid. At this stage the sol may still test negative, though at least traces of a nucleus will probably become discernible after some additional intervals. With further aging more fine black sediment slowly accumulates until it may cover the entire bottom of the flask. This becomes steadily more difficult to dislodge, even when the liquid is swirled vigorously, unless the sides and bottom of the flask are first scraped clean with a stirring rod. Long before that stage is reached the spot formed by the sol after stirring will have become definitely positive.

The shorter the negative period ultimately proves to be, the quicker will be the transition in successive spot tests from mere traces of a ring or nucleus to a clearly defined positive spot. Not long thereafter the appearance of the spot test will usually stabilize, remaining practically unchanged during the successive aging periods of the sol, as if a definite equilibrium had become established between its dispersing and flocculating components. This is illustrated in lines (*a*) and (*b*) of Fig. 1. In some cases, however, the nucleus may remain unchanged for only a limited period, but may then rather abruptly intensify and stabilize again in its new form, as illustrated in lines (*c*) and (*d*) of Fig. 1.

After the flocculate in any of the 39 sols has been thoroughly stirred in, it begins to settle again if left undisturbed, and continues to do so until the supernatant liquid becomes absolutely clear and forms a negative spot on filter paper, no matter how black a nucleus the drop from the well-stirred sol had previously produced (compare the spot of the clear and stirred liquid in the last two columns of Fig. 1).

The rate at which the supernatant liquid thus clears itself varies widely with different asphalts. Tests recently made on these sols by inserting a rod just under their surface at regular intervals after they had been well stirred and set aside indicate that the top stratum of the liquid will begin to test negative in as little as a half-day in the case of the Trinidad residuals (Items 18 and 19) whose flocculate, though small in amount, has always proved particularly hard to stir up, and in as much as ten weeks in the case of such long-negative-period asphalts as Smackover residual (Item 1). It is probably significant that when the sols of several asphalts from one source are compared, it is found that after being well stirred they resist clearing up in about the same order as they had resisted the development of heterogeneity; for example, the 10-yr old sols of Smackover residual and its two blends with 5 and 10 per cent of cracking-coil-tar No. 2 (Items 1, 34 and 35), which had taken respectively about 5, 3, and 1 yr to turn positive, took respectively 70, 30 and 14 days to clear up again after thorough stirring.

Simultaneously with the clearing up of the supernatant liquid, there is a corresponding concentration of the flocculated bodies towards the bottom of the flask. It will be observed that the rate of settling of this flocculate is very much faster than that at which it occurred in the freshly prepared spot test sol, so it must be evident that the flocculate in the aged medium no longer exists in the same form as it did originally, and that, among other possible changes, coarsening by agglomeration has no doubt occurred.

CHANGES IN INTERNAL STRUCTURE INVOLVED IN FLOCCULATION

This flocculate undoubtedly belongs, or is at least near-kin, to the intermediate (or B) group of asphaltenes[3] known as carbenoids,[3] since the latter term has been applied to all those asphaltene bodies that precipitate from the standard spot test sol. However, while ordinary carbenoids are usually expected to separate out within 24 hr, those that are capable of remaining dispersed for months and years must belong to a type of carbonoids more highly resistant to flocculation and nearer to the A group of the asphaltene family, which group is composed of the lightest, or rather the most easily dispersible, of the asphaltene bodies. Furthermore, the fact that these more-resistant carbenoids settle out a little at a time, and more slowly from the sols of some asphalts than from those of others, indicates that there must be a considerable difference in the degree of such resistance both as between the earlier- and the later-settling fractions of each flocculate, and as between the flocculates from each of the different sols.

Regardless of the many differences just pointed out between these more-dispersible carbenoids, which we would be justified in designating as carbenoids in only a potential sense, and regardless of the fact that as a group they resist flocculation better than ordinary carbenoids, they must all, like the latter, be classified as relatively coarse suspensoids —that is, suspensoids of coarser-than-colloid size and completely insoluble in their liquid medium; for if they actually were colloidal in size or were soluble in their medium, they would manifestly not have settled out of it within a matter of only days or weeks after they had once formed. And yet it must be evident that they could not have existed as such coarser-than-colloid dispersoids as they eventually became, either in the original asphalt or in the freshly prepared naphtha sol, for in the former case they would certainly have precipitated as a sludge whenever the asphalt was kept in a liquid condition for prolonged periods, as in storage, and in the second case they would have settled out of the freshly-prepared sol about as rapidly as they proved capable of doing after it had aged 10 yr.

Instead it must be assumed that originally these bodies existed in one or the other of the following two possible states: either as fine *colloidal* dispersoids in a medium in which, though inherently insoluble, they were able to stay permanently suspended by virtue of their colloidality alone, or as a complex system of micelles that are not really insoluble, since they *are* kept adequately dispersed or solvated in an equally complex system of solvating media and form with the latter a typical *gel* structure.

There no longer appears to be left much room for doubt as to which of these two systems is the most probable one. The subject has been discussed exhaustively by the author in a number of publications during the past 10 yr.[2, 3, 4, 5] All evidence that has been slowly building up in his researches since 1931, based on the study of such widely-diverse phenomena as those manifested by the spot test in dilute asphalt-naphtha sols on the one hand, and on the other hand by diffusion or sudation reactions between all types of bitumen in the semisolid state, cannot be reconciled with any other conclusion than that all bitumens are, without exception, essentially solvated and extremely complex gel-like products, whose equilibrium, or stability of internal phase relationship, is assured only when the solvating power of their various so-called "resinous" components is sufficient both to solvate adequately all their solid asphaltene micelles on the one hand and to neutralize the desolvating power of their more liquid "petrolene" components on the other. Should high heat or other analogous agents condense, or increase the solvation-resistance, of these micelles to the point where the solvating power of the resinous fraction becomes inadequate for its task, enough of the most condensed or more refractory of the asphaltenes will be flocculated until equilibrium within the rest of the system is restored. On the other hand, should enough petroleum naphtha be added to more than neutralize the excess solvating potency of the resinous phases, the same thing will occur again—namely, just enough of the least solvated fraction of the asphaltenes in the system will have to be forced out to enable equilibrium to be again restored.

As is characteristic of gel systems, the trend towards equilibrium is always relatively slow, and becomes progressively slower the closer to equilibrium they have attained. This is well illustrated in all the diffusion or sudation reactions studied by the author,[4] which invariably became noticeably retarded just before they ceased altogether. It is again illustrated in the present long-drawn-out series of tests with the naphtha sols of over 20 of the more highly homogeneous asphalts. Because of their higher homogeneity, their various phases must have been relatively close to equilibrium, with enough reserve solvating power in their resinous components to enable them in

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[3] G. L. Oliensis, "Reaction to 'Partial Solvents' as a Criterion of Structure in Bitumens," *Proceedings*, Assn. Asphalt Paving Technologists, Vol. 17, p. 1 (1948).

[4] G. L. Oliensis, "Gel Structure in Bitumens," *Journal of Asphalt Technology*, Vol. 4, No. 5 (1945).

[5] G. L. Oliensis, "Exudation and Allied Reactions Between Bitumens," Chapter 25 and 27, Chicago Bureau of Bituminous Technology, Forest Park, Ill., (1949.)

the early months of the sol's life to cope even with the desolvating forces of the excess quantity of naphtha present and greatly retard the latter's ultimate effects.

In these earlier months it can be assumed that the resinous phases exist as a series of protective layers around each of the micelles through which the naphtha-diluted petrolenes cannot immediately penetrate, and since the entire system is, as explained, still so close to equilibrium such further permeation will necessarily be slow; but if the ultimate desolvating power of the naphtha-diluted petrolenes should indeed be greater than the solvating potency of the resinous phases can cope with, then the system is *not* in satisfactory equilibrium any longer and the petrolenes will inevitably, though extremely slowly, start in motion that cycle of adsorption, diffusion and desolvating reactions that *will* restore equilibrium, the end result of which cycle will necessarily be a rejection of just enough of the least easily tolerated micelles in the system to accomplish that task. These will no doubt be precipitated, small portion by portion, over a period of months or years, in the order of their decreasing refractoriness, until the solvating force of the resinous components, though diluted and weakened by the added naphtha, again becomes capable of coping with the asphaltene phases still left and thereupon equilibrium is again restored.

The hexane reagent, both because it has an inherently stronger desolvating power than the standard spot-test naphtha, and because its addition to the sol still further dilutes and weakens the potency of the solvating components, no doubt disrupt the equilibrium of the system more completely than the spot-test naphtha already present, and hence forces a much more rapid rejection of the most poorly tolerated components to restore the equilibrium state. Hence it is not surprising that the addition of the hexane increments produces essentially the same effect in a matter of hours that the standard naphtha diluent exercises in a matter of months or years.

Any negative asphalt has its own characteristic equilibrium (or near-equilibrium) state—its own individual system of desolvated, solvating and desolvating phases that usually balance each other more or less adequately. Any change in its normal refining process that alters the character of any of these phases—for example, higher or lower than normal refining temperatures, the various effects of which have been discussed in another paper,[3] or any admixture with foreign asphalts of either a more or a less homogeneous type—for example, Gilsonite at one extreme and cracked asphalt at the other—is bound to alter that equilibrium state. That alteration may long remain concealed from view, but will inevitably be revealed by any testing procedure that can accelerate or intensify the readjustment within the altered system whereby some of its less tolerated components are forced out of it, either in the form of desolvated bodies as in the spot tests or of liquid exudates as in the sudation tests.[5] By comparing the speed of reaction, as well as the appearance and volume of the separated bodies resulting from any such test procedure on a given pair of asphalts, any deviation between the two is unerringly disclosed. This has already been demonstrated earlier in the present paper, but these concluding paragraphs give an insight into the series of changes in internal structure that produce the outward manifestations that make such comparisons possible.

CONCLUSION

The foregoing study supplements the author's 1941 paper[2] and verifies the conclusions previously drawn therefrom. It incidentally also supplements his thesis that the reactions of "partial solvents" on a bitumen are a criterion of its internal structure.[3] It serves to explain more completely than the 1941 paper the mechanism responsible for the very slow flocculation of near-carbenoids from negative asphalt sols. Finally, it reveals why a comparison of the rate and volume of such flocculation with the normal rate and volume enables the plant chemist or researcher to determine, with greater certainty than is possible by any other means except the sudation tests,[5] whether any asphalt, negative, positive, or intermediate, has deviated in any way from its "norm-," either through some abnormality in its refining process, (whether for the better or for the worse), or through blending with asphalts of other types, (whether better or worse), or through other abnormal conditions too numerous to mention.

DISCUSSION

MR. I. WEBER.[1]—In the author's opinion, can the resistance to checking and cracking of asphalts in weathering be correlated to the naphtha sol spot test?

MR. G. L. OLIENSIS (*author*).—Such a correlation cannot be established as definitely as might be wished, inasmuch as this paper has shown that there may be a marked difference in the behavior and stability of the spot-test sol of even carefully prepared and initially homogeneous asphalts, if those asphalts happen to be derived from different sources, and we unfortunately do not have available as yet sufficient data on the actual weathering characteristics of these various homogeneous asphalts under identical exposure conditions to prove how closely their service performance can be correlated with the behavior of their spot-test sols.

MR. WEBER.—I believe that it is generally recognized that cracked asphalts are much less resistant to weathering than the uncracked asphalts, and I thought there might be a possibility of correlation in the spot test to the weathering resistance.

MR. OLIENSIS.—It is true that there is now quite a formidable array of data pointing to such a trend as you mention; and insofar as a negative spot establishes that an asphalt has not been subjected to cracking, or does not contain an admixture of cracked asphalt, that test has a definite practical use to engineers and other consumers who have found that cracked asphalts do not give them the weathering service that they require.

But it is only fair to point out that asphalts are used at times for services in which resistance to weathering may not be involved, or where such characteristics as are peculiar to cracked asphalts may even be desirable. In that event the engineer or consumer would probably want to use the spot test to insure his obtaining an asphalt that *is* cracked.

However, it should not be overlooked that the primary function of the spot test is not the specific one of differentiating between cracked and uncracked asphalts but the more general one of differentiating between asphalts having a relatively unstable internal equilibrium (represented largely but by no means exclusively by the cracked asphalts) and asphalts having a relatively stable internal equilibrium (represented largely but by no means exclusively by the vacuum or steam-refined asphalts).

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[1] Ordnance Corps, Frankford Arsenal, Philadelphia, Pa.

Furthermore, the various modifications of the spot test make it extremely sensitive to small variations in that equilibrium, and since we have found that the latter can be disturbed by small changes in the methods of refining or by even minor adulterations that may alter the character of an asphalt, the spot test becomes a tool for insuring extreme uniformity in the quality of *any* given type of asphalt—regardless of whether it is cracked or uncracked, or has good or bad weatherability, or superior or inferior serviceability in any other direction. Hence the utility of the spot test covers a much wider and somewhat different field from the concrete one you have mentioned of differentiating between asphalts that weather well and asphalts that do not.

PROPERTIES OF EXPOSED AND UNEXPOSED POLYVINYL BUTYRAL COATED FABRICS*

By Meyer I. Landsberg,[1] Thomas J. DiFilippo,[1] and Ladislav Boor[1]

Synopsis

A series of polyvinyl butyral resins, plasticized with varying amounts of approximately equal parts of trioctyl phosphate and dibutyl "cellosolve" sebacate, were evaluated both as thick slabs (0.05 to 0.075 in. thick) and when coated on 1.6 oz per sq yd nylon. These materials were evaluated for their physical properties in the original and exposed states, using standard and non-standard laboratory tests and for plasticizer and phosphorus content, employing standard analytical techniques.

The physical evaluations indicated that the most important variable in its effect on the coated fabric was the modulus of the coating compound in its final cured form.

Data are presented to show the effect of Florida exposure on such properties as tearing strength and flexibility. It is shown that approximately 360 ultraviolet hrs of exposure are required to degrade these materials seriously.

Plasticizer and phosphorus ratio determinations on these materials indicated that in this plasticizer system, the dibutyl "cellosolve" sebacate is lost at a much faster rate than the trioctyl phosphate and also that the ultimate loss of the former is much greater than that of the latter. Finally, it was ascertained that the ultimate loss of the total plasticizer is in the order of 60 per cent after 1440 sun-hours of exposure. Possible reasons for the differential loss of plasticizer are advanced.

In an effort to achieve suitable lightweight rainwear which would have desirable tear, flexibility, low-temperature and degradation resistance characteristics, a series of coated fabrics was prepared in which the coatings were varied in a systematic manner. The base fabric in all instances was 1.6 oz per sq yd nylon, selected and tested for uniformity of tear strength. The coating resin was polyvinyl butyral, plasticized with varying amounts of plasticizer consisting of equal parts of trioctyl phosphate (TOF) and dibutyl "cellosolve" sebacate (DBCS). The finished coating contained 40, 70, 100, 130, 150, and 200 per cent plasticizer, respectively, based on the resin content. These materials when applied to the nylon base produced a series of coated fabrics having from 4 to 18 layers of plasticized resin. Some of the fabrics had the same number of coatings on both sides (balanced); and others had more on one side (unbalanced). In addition, the coating compound was also made into slabs approximately 50 to 75 mils thick. The slabs and fabrics were cured in an analogous manner to provide both a coating composition in slab form and a fabric coated with the same composition. Thus, evaluation of both the coated fabrics and the slab-like coating, in the original and exposed states, would furnish data concerning the effects of (1) per cent plasticizer, (2) balance and unbalance, (3) number of coats of resin, (4) rheological properties (stress-strain) relationships of the coating itself, (5) degree of penetration and adhesion on the physical properties of the coated fabric.

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* Presented at the Fifty-Fourth Annual Meeting of the Society, June 18–22, 1951.

[1] Quartermaster Research and Development Labs, Chemicals and Plastics Section, Philadelphia, Quartermaster Depot, Philadelphia, Pa.

Concurrent with the physical tests, a number of chemical determinations were made to assist in better understanding the rôle played by the plasticizers in the material. Determination of the per cent plasticizer extracted from the exposed and unexposed specimens and phosphorus analyses of the extracted plasticizer permitted certain conclusions to be reached concerning the effect of Florida exposure on total plasticizer loss, as well as loss of each of the two plasticizer components (TOF and DBCS).

EXPERIMENTAL PROCEDURE

Processing:

All of the fabrics were knife-on-roll coated. Most of the solvent (alcohol) was driven off by open steam coils maintained at a temperature of about 240 F. The temperature of the fabric varied between 150 F and 180 F during this operation. The material was cured in a festoon oven for 1 hr up to 250 F (as material was being heated up) and then for 1 hr at 250 F to 260 F, at which point the cure was complete.

Sun Exposure:

The fabrics evaluated in this study were tested in their original condition and after exposure. The specimens were exposed simultaneously in Miami, Fla., on a series of 45-deg racks, facing south. The recording instruments were mounted in a like manner. Daily records were kept of minimum and maximum temperatures, relative humidity, extent and intensity of rainfall, total sun-hours, total ultra-violet hours and Btu's per sq ft during U. V. sun hr. Eppley Pyrheliometers and Leeds & Northrup Special Micromax curve-drawing recorders were employed to measure solar radiation. The specimens were exposed for periods of 180, 360, 720, and 1440 hr of ultraviolet radiation.

Test Methods:

Physical.—The selection of the physical test methods for evaluating the fabrics was based on the need for two types of data. One group of data desired was that which could be used to set specification levels when the need arose; the other was to be used for determining more fundamental properties of the coating in slab form, and of the fabric-coating combination. To achieve the former, such tests as (1) hydrostatic pressure (Mullen) (**1**),[2] tearing strength (Elmendorf) (**2**), impact tear (**3**), and stiffness (Clark) (**4**), were conducted. In the latter group, the Instron Tensile Tester (**5**, **6**) a high-precision electronic tensile testing machine, was used to determine the stress-strain properties of the coating substances made in slab form. The flexural and torsional moduli were determined on the Tinius Olsen (**7**) and Clash-Berg Instruments (**8**), respectively.

Chemical.—First, plasticizer determinations were made using a solvent extraction method. A 3-g sample (approximate) of the coated fabric was extracted for a period of 5 hr with petroleum ether, after which the ether was driven off, the extracts were oven-dried at 150 F, then desiccated under vacuum, and, finally, re-weighed. Progressive plasticizer loss could then be related to physical properties. This was only a partial answer, since it was still not possible to determine the relative loss of the individual component.

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[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1275.

The next step was to determine the per cent phosphorus in the residual plasticizer. Analysis of a 50:50 mixture of TOF and DBCS indicated the presence of 3.51 per cent phosphorus. Phosphorus was determined by digesting the plasticizers with concentrated sulfuric acid and hydrogen peroxide, using a selenium catalyst. After digestion, concentrated nitric acid and distilled water were added to make up a known volume. Aliquot portions were removed, treated with ammonium molybdate, and titrated with sodium hydroxide past the end point. Finally, they were back-titrated with nitric acid.

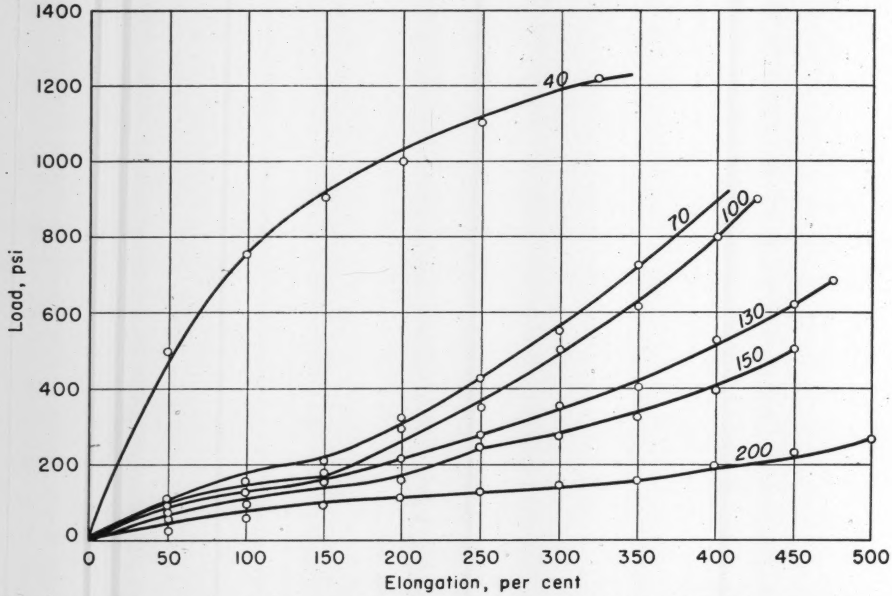

Fig. 1.—Stress-Strain Curves for Various Polyvinyl Butyral Resin-Plasticizers Systems.

PHYSICAL TEST DATA

First, physical data were obtained on both the coating substance alone and on the coated fabric. Using these data, an attempt was made to correlate such variables as number of coats, balance of coats, weight and amount of plasticizer with breaking strength, flexibility, and tearing strength. The only correlation obtained was between amount of plasticizer and the results of the above-mentioned laboratory tests. To evaluate properly the rôle played by the coating alone, correlations were established between coating substance properties and amount of plasticizer. Next, relationships were established between coated fabric properties and amount of plasticizer. Finally, relationships between coating substance and coated fabric properties furnished some information as to relative importance of the coating alone and the fabric to which it was applied. For example, no relationship was evident between amount of plasticizer and breaking strength of the coated fabric. This was understandable in view of the fact that breaking strength of a coated fabric is primarily a fabric function and not a coating function. However, establishment of a relationship between the breaking strength of the coating and amount of plasticizer furnished valuable information as to the part played by the coating.

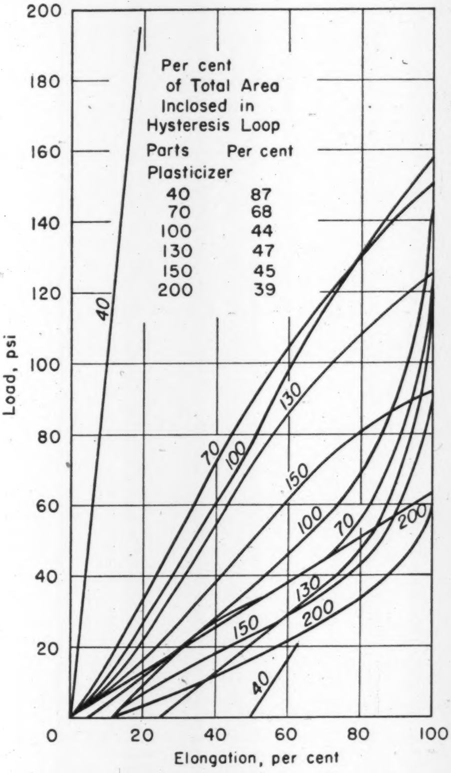

FIG. 2.—Hysteresis Curves for Various Polyvinyl Butyral Resin-Plasticizer Systems.

The various relationships which were obtained indicated: (*a*) the most important variable, in its effect on the coated fabric, is the modulus[3] of the coating compound in its final form, and (*b*) most of the significant properties of the coated fabric varied only insofar as the modulus of the coating varied.

Coating Compositions:

Evaluation of the coating compositions, molded into slabs approximately 0.050 to 0.075 in. thick, permitted segregation of the effects of coating alone. With the exception of a single anomalous result on the sample containing 70 parts plasticizer per 100 parts of resin, there is nearly an inverse straight-line relationship between breaking strength and amount of plasticizer. The rate of decrease in this property is very rapid, with the specimen containing 200 parts plasticizer having approximately one-quarter the breaking strength of the one with 40 parts.

Concomitant with the tensile measurements, per cent elongation at the break was also obtained. These results, when plotted against per cent plasticizer yielded a curve showing approximately a two-fold increase in extensibility in going from the least to the most extensible material.

In addition to the two previously mentioned tests, stress-strain curves on the slab materials were also obtained on the Instron Tensile Tester. Figure 1 shows the stress-strain curves obtained on the series at 5 in. per min jaw separation. The load elongation values for the various slabs fall into proper sequence, namely, that the slabs with the least amounts of plasticizer elongate less and have a higher breaking strength at the point of failure. In addition, at any one elongation, the tensile stress is greater for the low-plasticizer content samples than for the higher. The curves for the higher plasticizer content samples show a reversal of slope at approximately 150 per cent elongation, but not the curve for the 40 parts plasticizer sample. The curves for the compounds made with 150 and 200 parts plasticizer are somewhat similar to those which are obtained for certain types of rubber.

The effect of loading and unloading

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[3] Modulus is used here not in the strict physical sense of the slope of the elastic-stress strain relationships, but rather as an index of resistance to deformation.

to 100 per cent elongation at 5 in. per min jaw separation is presented in Fig. 2. The decrease in proportion of the hysteresis loop as plasticizer is increased, and the fact that the formulations containing 100 to 200 parts plasticizer retract to within 12 per cent of their original length, indicates that such coating substances recover deformations (such as folds and creases) almost completely.

To further utilize basic data on coating compositions which could be used to achieve desirable coated fabrics for Army use, the tensional modulus was obtained of polyvinyl butyral plasticized resin in slab form at various degrees of extension as shown in Fig. 3. The curve for the 40 parts plasticizer shows a sharp drop of modulus indicating a high degree of time-sensitiveness, tendency to creep, and poor recovery. It is indicated from this relationship that fabrics coated with compounds containing 130 to 150 parts plasticizer would have good resilience and recover creases almost instantaneously, as compared with the low plasticizer coatings which retain packing and folding creases almost indefinitely. Although the 200 parts plasticized resin has excellent recovery properties, its tacky feel and low abrasion resistance preclude its use as a coating for fabric intended for rainwear.

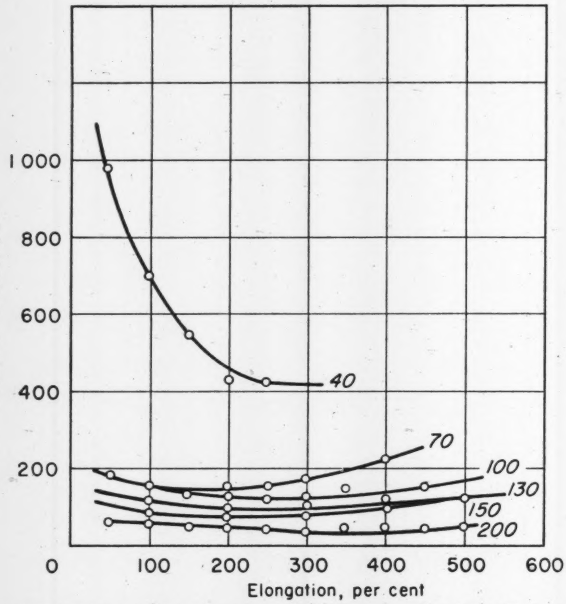

FIG. 3.—Modulus of Plasticized Polyvinyl Butyral Resin at Various Degrees of Extension.

Flexural modulus at 5-deg angular deflection (Tinius Olsen Stiffness Tester) plotted against temperature is shown in Fig. 4. The important rôle of plasticizer content is evident. The lower plasticized coatings (40 and 70 parts) show large changes in modulus with only slight temperature decreases. The 130, 150, and 200 parts plasticized coatings give fairly good flexibility even at temperatures of −40 F. In fact, the modulus of the 40 parts plasticized sample is only slightly lower at room temperature than that of the 150 plasticized sample at −20 F.

The graph shown in Fig. 4 served to substantiate the importance of the modulus of the coating. It appears that there is sufficient evidence, to date, to show that modulus is one of the most important factors in determining coating characteristics. Although the general concept regarding modulus must still be extended to other coating resins and elastomers, there appears to be available a tentative working basis which can serve to predict trends. The curves in Fig. 5, in which moduli of torsional rigidity, as determined by the Clash-Berg method, are plotted against temperature, compare quite well with similar curves obtained by other investigators (9) on polyvinyl chloride films, thus indicating the similarity of the polyvinyl butyral resin system to other resin-plasticizer systems.

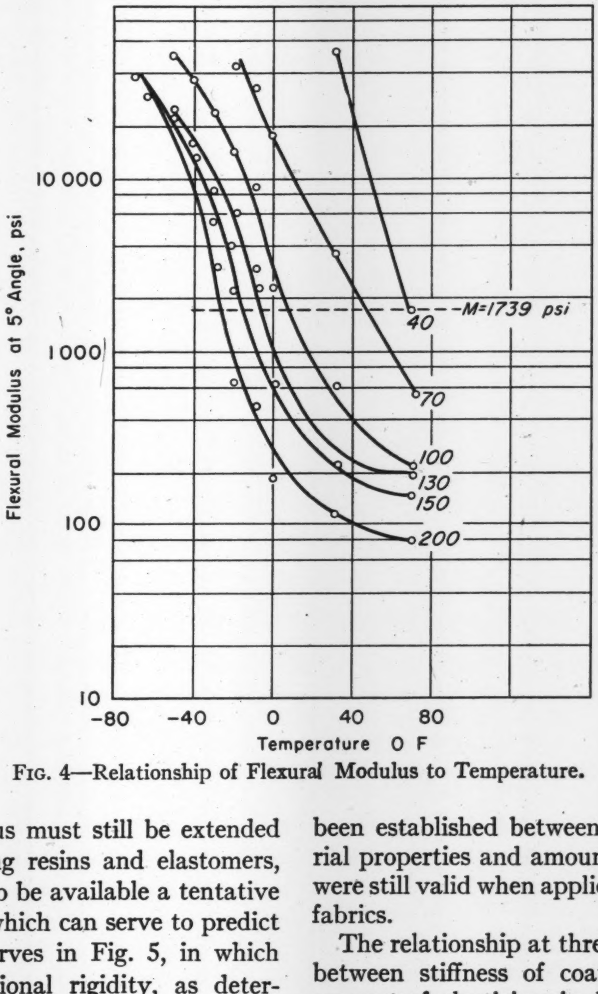

FIG. 4—Relationship of Flexural Modulus to Temperature.

Coated Fabrics:

The next step was to evaluate the coated fabric and ascertain whether the same general relationships which had been established between coating material properties and amount of plasticizer were still valid when applied to the coated fabrics.

The relationship at three temperatures between stiffness of coated fabric and amount of plasticizer is shown in Fig. 6. The points representing the various amounts of plasticizer are the averages obtained on all the samples irrespective of coating weight, balance, or number of coats. It can be seen that stiffness is af-

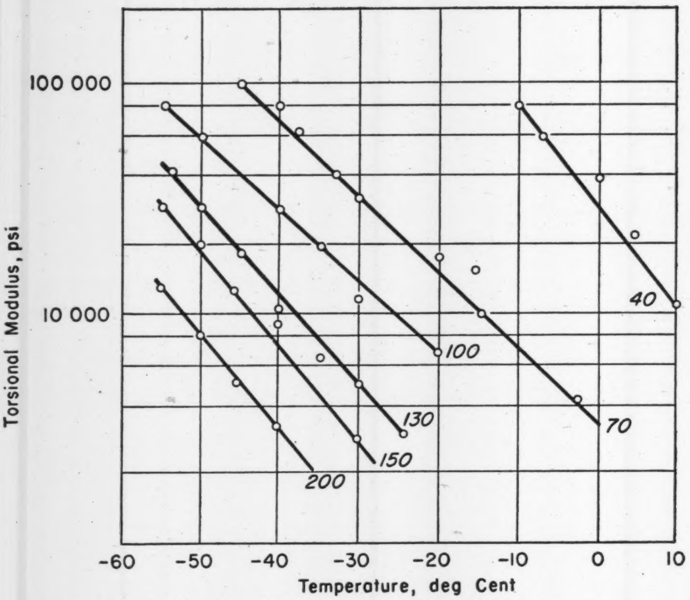

FIG. 5—Relationship of Torsional Modulus to Temperature.

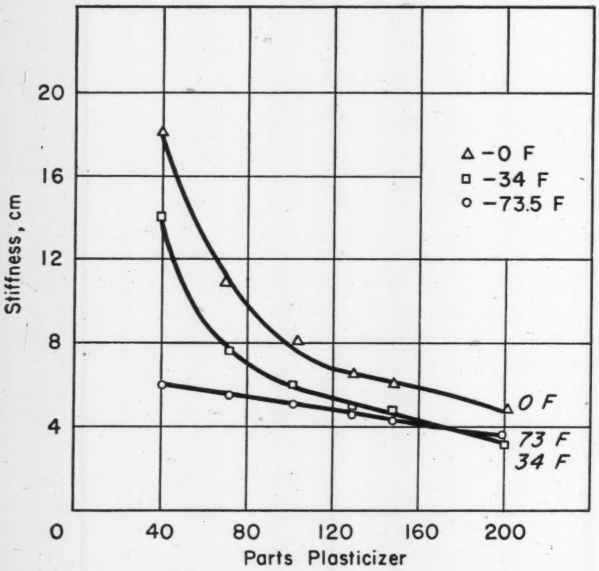

FIG. 6—Relationship of Clark Stiffness to Parts Plasticizer.

fected by both plasticizer content and temperature. As temperature and plasticizer content decrease, the stiffness increases. Since stiffness is related to modulus, comparison of flexural modulus of the coating to stiffness of the coated fabric permits quantitative assessment of the rôle played by the modulus.

Comparison of tensional and flexural moduli of coating substance with stiffness of coated fabric verifies the commonly accepted relationship between them.

Considering all the relationships described, it appears that the specification tests commonly required, such as stiffness or tearing strength, are related to more fundamental coating substance properties. The fact that the commonly used laboratory tests measure these properties adequately, makes it possible to use these methods to set up realistic acceptance values for specification purposes.

It has been previously shown that both temperature and amount of plasticizer affect the modulus of the coating material. It was therefore desired to ascertain the extent of this influence on the coating-fabric combination. Flexural modulus (Tinius Olsen Stiffness) was obtained on the unexposed specimens at 73 F, 32 F, 0 F, and −20 F. It was found that as amount of plasticizer increases, the modulus decreases. A decrease in temperature also causes large increases in modulus. In general, it is indicated that the influence of temperature is similar on both the coating and on the coated fabric. These concepts are not new, but the quantitative relationships obtained on the coated fabrics yielded clues as to what systems are essential to provide adequate flexibility at low temperatures.

It was felt that one other experiment on the coated fabric, using data obtained on the coating alone, should verify the importance of modulus and the part it assumes in determining desirable coated fabric characteristics. The modulus of the lowest plasticizer content (40 parts) was used as the starting point, and the dotted line in Fig. 4 was then drawn through the family of curves as shown. The intersection of the dotted line with the solid curves shows the point on each curve where the various temperatures produce the same modulus. The tearing strength was determined for each of the specimens at the indicated temperatures. The results (Table I) show that nearly identical tear strengths are obtained on all of the fabrics when each is cooled to a temperature at which their respective moduli are comparable. Thus it is indicated that, given sufficient data on other resin-plasticizer systems, it should be possible to predict the behavior of a coated fabric when certain fundamental properties of the coating are known.

TABLE I.—MODULUS VERSUS TEAR STRENGTH OF ORIGINAL MATERIAL.

Parts Plasticizer	Flexural Modulus at 5 deg Angle	Elmendorf Tear Energy, in-lb. per in.	Temperature, deg Fahr	Flexural Modulus at 5 deg Angle	Elmendorf Tear Energy, in-lb per in.
	(Coating) 73 F	(Fabric) 73 F		(Coating) At respective temperatures	(Fabric)
40......	1739	3.1	73.5	1739	3.0
70......	536	5.3	42.0	1739	3.4
100......	208	5.8	4.0	1739	3.4
130......	208	7.1	−6.0	1739	3.4
150......	143	13.8	−10.0	1739	3.5
200......	83	15.0	−28.0	1739	3.8

It should be borne in mind that the term "modulus" does not imply a fixed physical constant as is commonly used in describing the behavior of metals. In materials such as those described, the response to stressing is enormously affected by the rate of stressing, and in that sense, the dimension of time.

It had been previously established that the coatings containing 130 to 150 parts plasticizer appeared to offer the best possibilities as military fabrics; therefore the abrasion resistance was of interest. To ascertain the relative resistance to abrasion of the various polyvinyl butyral, formulations, a series of tests were made on the Stoll Abrasion Tester (10). The specimen was abraded until a sufficient amount of coating had been removed to expose a small uniform area of fabric. Evaluation of a large number of specimens of various thicknesses provided data of statistical validity for further computations. The number of rubs (to expose the fabric) divided by the thickness of coating provided an index of abrasion which is designated as rubs per mil. The relationship between rubs per mil and nominal plasticizer content is shown in Fig. 7. Although abrasion resistance decreases with increasing plasticizer content, the coatings containing 130 to 150 parts plasticizer appear to have sufficient abrasion resistance to warrant their consideration in Army items, particularly in view of their excellent tear and flexibility characteristics at all temperatures.

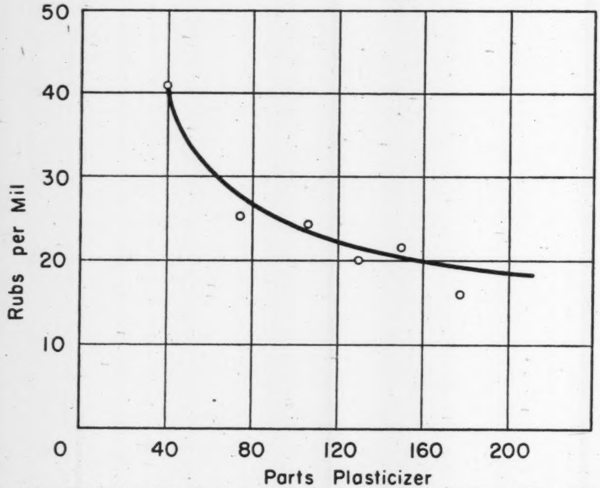

FIG. 7—Relationship Between Rubs per Mil and Parts Plasticizer.

Deterioration by Outdoor Exposure:

During the exposure periods of 180, 360, 720, and 1440 ultraviolet hrs, the fabrics are subjected to certain uncontrollable variables such as rainfall, relative humidity, temperature, and Btu per sq ft. The differences in these variables, from one exposure period to the next, may cause the variations in test data. For example, during the first exposure period (0 to 180 ultraviolet hr), the samples received 303 Btu per sq ft during each ultraviolet sun-hour; they were rained on to the extent of 0.050 in. per hr, received 327.0 hr of total sun, and subjected to maximum and minimum temperatures of 81 F and 65 F, and 95 per cent and 57 per cent relative humidity, respectively. During another interval of exposure (720 to 1440 ultraviolet hr) the samples received 277 Btu per sq ft; they were rained on to the extent of 0.040 in. per hr, and subjected to maximum and minimum temperatures and relative humidities of 94 F and 39 F, and 100 per cent and 19 per cent, respectively. Notwithstanding variations, it was found that the degradation of samples during each exposure period was still sufficiently uniform to permit rather smooth curves to be drawn.

The effect of Florida exposure on stiffness is shown in Fig. 8. Up to 180 hr, very little stiffening occurs, but at the end of 360 hr the unbalanced coatings, the heavily coated side of which were exposed to the sun, show large losses in flexibility. The balanced coatings, on the other hand, show only slight losses in flexibility. The large differences in stiffness between the balanced and unbalanced coatings are probably due to the mechanical stiffening of a thick layer (unbalanced coating) as compared to two thin layers separated by the nylon fabric. At 1440 U.V. hr, it can be noted that the sample containing 40 parts plasticizer is less stiff than the the one containing 70 parts plasticizer. This lowered stiffness at 1440 U.V. hr is attributed to the cracking of the coating.

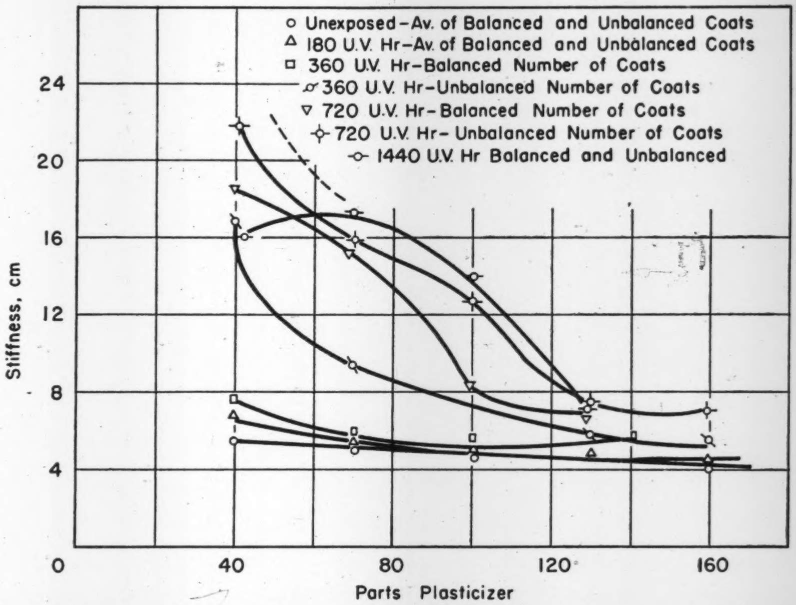

FIG. 8—Relationship Between Stiffness and Parts Plasticizer Originally and After Exposure.

The change in tear energy with exposure is shown in Fig. 9. As in the relationship between stiffness and amount of plasticizer, the amount of degradation becomes severe at 360 sun-hours, after which, the rate decreases. For specification requirements, the ability of a coated fabric to withstand 360 U.V. hr of exposure should make it satisfactory from a use standpoint.

That the coatings represented by this series of fabrics stiffen during Florida exposure has been verified, but the exact cause for the stiffening has not been determined. It has also been accepted, generally, that plasticizer loss is a contributing factor, but how plasticizer is lost is not clear. Fungi and bacteria have previously been found to be active in destroying oil-type plasticizers (11). There is also evidence that ultraviolet light, ozone, moisture and oxygen play a rôle in the chemical or physical loss of plasticizer. Therefore, a few preliminary experiments were carried out in the laboratory to determine the probable causes of stiffening (plasticizer loss). Specimens similar to those exposed in Florida were subjected to the following simulated weathering procedures in the laboratory:

(*a*) S4 Sun Lamp.
(*b*) National Weathering Unit X1A Unit.
(*c*) Water leach (running water at 70 F).
(*d*) Alternate water leach (16 hr) and sun lamp (7 hr).

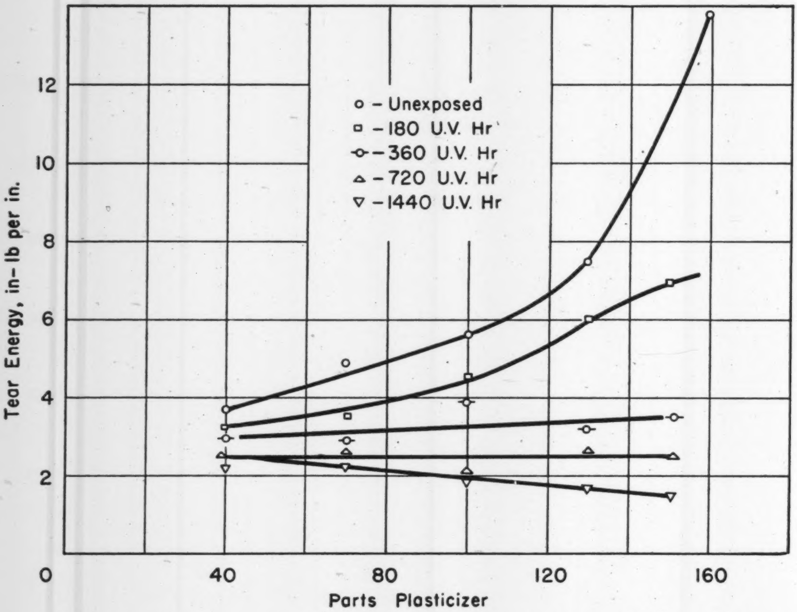

FIG. 9—Relationship Between Tear Energy (Elmendorf) and Parts Plasticizer Before and After Exposure.

The sun lamp alone caused practically no change in stiffness up to 367 hr. Both the Weatherometer and water leach alone produced the same stiffening at approximately 300 hr. At about 900 hr of water leaching alone, and at 400 hr of water leaching and 176 hr of sun lamp exposure, the stiffening of the 40 and 70 parts plasticized samples were of about the same order. The stiffening, at these exposures, was quite severe. It appears, therefore, that the greatest degrading effect is caused by the combination of water leaching and sun lamp.

The verification of these effects will be held in abeyance until such time as a newly designed Weatherometer, which can control both relative humidity and temperature within narrow limits, is constructed (12).

CHEMICAL TEST DATA

The determination of plasticizer and phosphorus by the methods described under Test Methods: Chemical, did not present any undue difficulties, but several modifications had to be made in the method (13) of determining plasticizer content to insure reproducible results. First, it was determined that a 5-hr extraction period was required to remove all the plasticizer.

To confirm the completeness of plasticizer removal from the fabric, phosphorus determinations were made on the residue after normal extraction. The phosphorus remaining ranged from 0.0057 per cent to 0.0081 per cent.

Having established that the plasticizer is removed quite completely, a second experiment was carried out to ascertain the time required for the petroleum ether, used in the extraction, to be almost completely removed. Based on this experiment, it was decided to select a $2\frac{1}{2}$-hr heating period as being most suitable.

Having established the proper conditions of test, plasticizer and phosphorus determinations were made as previously described, under Test Methods: Chemical. Each plasticizer value is the average of at least two determinations, and each phosphorus value, the average of at least four determinations. The results are shown in Table II.

TABLE II.—PLASTICIZER AND PHOSPHORUS CONTENT OF PVB COATED NYLON FABRIC, ORIGINALLY AND AFTER FLORIDA EXPOSURE.

Sample	N. P. P.[a]	Parts Plasticizer at Various Ultraviolet hr.					Per cent Phosphorus at Various Ultraviolet hr.				
		Orig.	180 hr	360 hr	720 hr	1440 hr	Orig	180 hr	360 hr	720 hr	1440 hr
No. 6579C	40	41.2	35.6	32.2	17.0	15.3	2.87	3.09	3.43	5.77	5.27
No. 6580B	40	42.1	30.8	17.8	16.4	17.3	2.77	3.32	5.68	5.50	4.85
No. 6581C	70	63.1	55.8	39.9	27.7	23.7	3.10	3.58	5.07	5.17	5.88
No. 6582B	70	69.6	38.2	27.3	25.1	22.8	2.48	3.25	6.51	5.31	5.55
No. 6583C	100	88.5	78.2	45.8	43.0	37.8	3.04	3.52	5.52	6.04	5.20
No. 6584B	100	85.0	64.8	38.9	36.2	29.0	3.17	3.40	5.36	4.44	5.84
No. 6585C	130	122.4	107.0	66.0	63.2	54.2	2.82	3.09	5.25	6.16	5.01
No. 6586B	130	131.4	108.2	54.3	53.5	48.8	3.01	3.60	6.09	4.72	5.27
No. 6659A	150	136.1	125.8	73.5	72.2	64.6	2.69	2.77	5.26	5.97	5.11

[a] Nominal parts plasticizer; per 100 parts resin.

The amounts of plasticizer shown in this table are based on the original weights of the resin in the coating compound, and the percentages of phosphorus are based on the weights of the extracted plasticizer.

Comparison of the nominal amount of plasticizer (per cent plasticizer mixed by processor into resin) with the amount of plasticizer found by analysis on the unexposed specimens shows that, in general, there was less plasticizer present than had been indicated by the processor. It is possible that the coating mix was properly formulated but that losses were incurred during processing.

Comparison of amount of plasticizer before and after exposure on all of the samples shows the same general trend: namely, that Florida exposure removes appreciable quantities of plasticizer. With the exception of samples 6579C and 6581C, the maximum loss occurred at the end of 360 sun-hours of exposure. Maximum loss in plasticizer content on the other two samples occurs after 720 sun-hours. Although the reason for this discrepancy is not known, it is possible that positioning effects during exposure, or microbiological attack, could cause anomalous results. It had also been assumed that the two plasticizers were present in the coatings in equal parts; however, phosphorus determinations (on the extracted plasticizer) from which per cent TOF was calculated and per cent DBCS found by difference, did not verify this. It was found that the proportion of DBCS to TOF in the original unexposed materials was not constant but varied from 70.6 per cent DBCS and 29.4 per cent TOF to 55.2 per cent DBCS and 44.8 TOF.

It can be seen from Fig. 10 that the proportion of original plasticizer lost is approximately the same for all samples. It is also evident that the weight of plasticizer lost is a function of the amount of original plasticizer present in the samples. After 1440 hr of exposure, the samples containing 41 parts and 136 parts plasticizer, respectively, lost 60 and 63 per cent plasticizer respectively, but the total weight lost was greater in the case of the sample with the higher plasticizer content.

The data also point to the fact that the DBCS is lost at a much faster rate than is the TOF. Whereas, at the end of the longest exposure period (1440 hr) only from 19 to 38 per cent TOF has been lost, from 79 to 89 per cent DBCS has been lost in this period of time. On the basis of the data presented, it can be concluded that the reason for loss in flexibility and tear resistance is the absence of DBCS. It is not possible at this point to account fully for the differential loss of DBCS and TOF, but possible contributing factors may be:

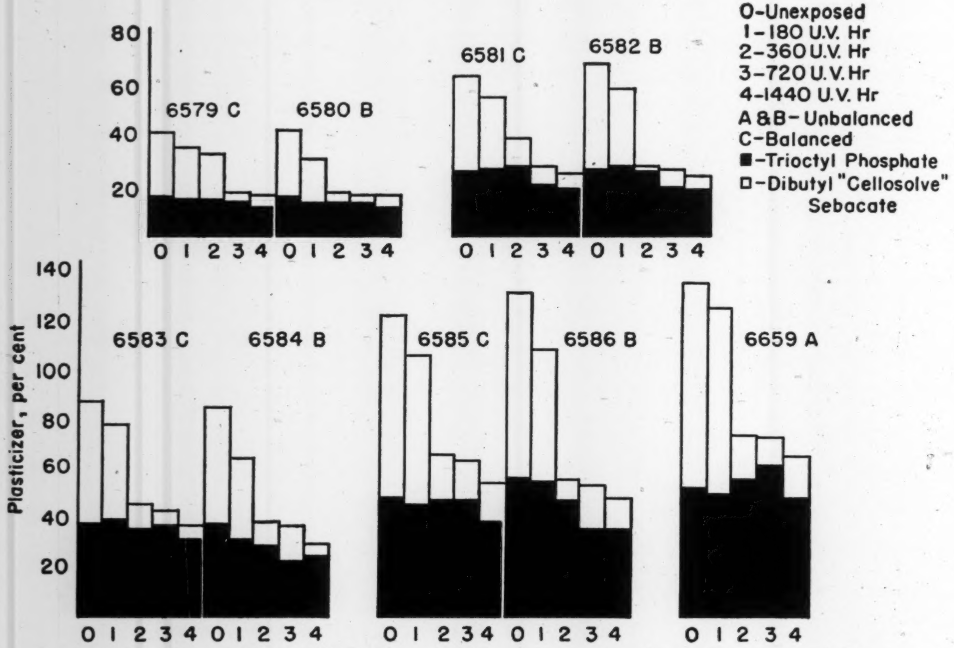

FIG. 10—Per Cents DBCS and TOF in Plasticizer Extraction from Exposed and Unexposed Polyvinyl Butyral Coated Fabrics.

1. Differential evaporation or water leaching rate from the film. Data in the literature appear to verify this (14, 15, 16).
2. Removal of sebacate by action of microorganisms.

It is known that phosphates are relatively resistant to attack by molds and fungi, but sebacates are vigorously attacked. In formulations containing 100 parts or more total plasticizer, removal of the sebacate still leaves enough phosphate to impart a fair degree of flexibility at room temperature; however, such a coating would show severe stiffening at low temperature.

In an attempt to confirm the part played by the various deteriorative factors in causing stiffening due to chemical alterations of resin or plasticizer, a series of experiments was undertaken. In these experiments, described more fully by Boor in a recent paper (12), two of the PVB coated nylons, containing 40 and 70 parts plasticizer respectively, were prepared in strip form and exposed (*a*) in quartz tubes, and (*b*) in glass tubes shielded by aluminum tubing, respectively. A set of each of the above was sterilized and exposed in the sterile condition. Another set, after sterilization, was inoculated with *Pseudomonas Aeruginosa*, a plasticizer-decomposing bacteria and also exposed. After a two-month exposure period (approximately 235 ultraviolet hours) the strips were tested for stiffness. They were again sterilized, reinoculated, and exposed for four more months (approximately 471 ultraviolet hours). At the end of this period, they were re-evaluated for stiffness.

According to these results, there appears to be no significant difference between the sterile and the inoculated samples, whether exposed under quartz or in the dark. A significant difference in stiffness is apparent between the specimens exposed in the dark and those exposed under quartz. Visual examination of the specimens reveals that, in the case of the inoculated samples exposed in the dark, there are a series of spots in the middle of, and running the whole length of, the specimens. These spots appear to have been caused by bacterial attack, initiated at the areas where drops of the inoculating broth had dried. It was noted that in this area the coating does not recover from scratch marks, when scratched with a needle, as does the coating in the surrounding areas. Also, ends of the test strip where the inoculum (bacteria in broth) had concentrated and dried were severely stiffened. Indications are, that areas where bacterial growth was most active had stiffened more than the surrounding, less severely attacked areas.

On the basis of the data obtained, it can be concluded that sunlight exerts a greater effect on plasticizer loss than does microbiological attack, but there seems to be some evidence which points to the fact that plasticizer losses, as a result of both ultraviolet and fungal attack, are occurring simultaneously. In the case of the specimens tested, the area of attack was relatively small, and the local stiffening was overshadowed by the general over-all stiffening.

Acknowledgment:

The assistance of the Hodgman Rubber Co. and Monsanto Chemical Co. in making this work possible is gratefully acknowledged.

REFERENCES

(1) Federal Specification CCC-T-191 and Supplement, Section IV, part 5, p. 21.

(2) Federal Specification CCC-T-191 and Supplement, Section IV, part 5, p. 4.

(3) Impact Tear Test, Jeffersonville QM Depot Report No. 325, Feb. 1947.

(4) Federal Specification CCC-T-191 and Supplement, Section OV, part 5, p. 11.

(5) Harold Hindman and G. S. Burr, "The Instron Tensile Tester," *Transactions*, Am. Soc. Mechanical Engrs., pp. 789–796, Oct. 1949.

(6) G. S. Burr, "Servo-Controlled Tensile Strength Tester," *Electronics*, pp. 101–105, May, 1949.

(7) Tentative Method of Tests for Stiffness in Flexure of Plastics (D 747 – 487), 1949 Book of ASTM Standards, Part 6, p. 578.

(8) R. F. Clash, Jr. and R. M. Berg, "Vinyl Elastomers, Low-Temperature Flexibility Behavior," *Industrial and Engineering Chemistry*, Vol. 34, pp. 1219–1222 (1942).

(9) G. J. Dienes, "Viscoelastic Properties of Thermoplastics at Elevated Temperature," *Journal of Colloid Science*, Vol. 2, p. 131, Feb. 1947.

(10) R. G. Stoll, "An Improved Multi-Purpose Abrasion Tester and Its Application for the Evaluation of Wear Resistance of Textiles," R. G. Stoll, *Textile Research Journal*, Vol. 19, p. 394, July, 1949.

(11) "Studies on Degradation of Plastic Films by Fungi and Bacteria," J. V. Harvey and F. A. Meloro, PQMD, General Labs, Microbiology Services No. 16, August 15, 1949.

(12) Ladislav Boor, "Developments in Coated Facrics and Films by the Quartermaster Corps," QM Research and Development Lab; Chemicals and Plastics Section, Research Service Test Report C&P-150-F&CF, August 16, 1950.

(13) Determination of Plasticizer, Victory Plastics Co. Report.

(14) M. C. Reed and L. Connor, "Behavior of Plasticizers in Vinyl Chloride-Acetate Resins," *Industrial and Engineering Chemistry*, Vol. 40, No. 8, pp. 1414–1422, August 8, 1948.

(15) M. C. Reed, "Behavior of Plasticizers in Vinyl-Chloride Acetate Films," *Industrial and Engineering Chemistry*, Vol. 35, Part I, p. 896, August 1943.

(16) M. C. Reed and James Harding, "Plasticizers in Vinyl-Chloride Acetate Resins," *Industrial and Engineering Chemistry* Vol. 41, Part III, p. 675, April, 1949.

DISCUSSION

MR. ROBERT M. BERG[1].—I should like to ask the authors whether those exposures in Florida were all conducted simultaneously or did one group go down to Florida in October and another group in December, and so forth?

MR. M. I. LANDSBERG (*author*).—All of the Florida exposures were made simultaneously.

MR. BERG.—That is good, for we have learned that unless samples are exposed simultaneously, comparisons may be quite misleading. It is possible to send down a series of samples, say in November or December, and in a specified number of "ultraviolet sun-hours" get a certain amount of degradation; and then send down some more of the same samples the following June or July, for example, and after the same number of "ultraviolet sun-hours" find that the samples will have degraded to a considerably different extent than did the original series.

MR. LANDSBERG.—That is true. In this particular case all the samples were sent down at the same time. At the end of the first exposure period (180 ultraviolet hours) a set of samples was removed for test. Additional samples were removed at 360, 720 and 1440 ultraviolet hours.

I might point out that although there were some differences in the conditions (relative humidity, temperature, rainfall, and Btu's per sq ft during sunlight) during each of the exposure periods, we were quite surprised that the materials still seemed to show relatively uniform degradation over the total period of time.

The weather data obtained during Florida exposure has been summarized in Research Service Test Report C&P-190-CF under the same title as the present paper. This more comprehensive report can be obtained by writing to these laboratories.

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[1] Resins Lab., Carbide and Carbon Chemicals Co., Division of Union Carbide and Carbon Corp., South Charleston, W. Va.

CREEP RELAXATION RELATIONS FOR STYRENE AND ACRYLIC PLASTICS*

By Joseph Marin[1] and Yoh-Han Pao[1]

Synopsis

In addition to the usual constant tensile stress creep-time relations, this report gives tension creep relaxation test results for styrene and acrylic plastics. Stress-time relaxation curves are presented for various initial stress values. The relaxation relations were obtained for the usual condition where the specimen gage length did not change with time—that is, the strain remained constant. Based on the ordinary tensile creep-time relations—for various constant stress values—theoretical stress-time relations for the relaxation condition were predicted using various theoretical interpretations. It was found that a "strain hardening method" for predicting the relaxation curves agreed well with the actual relaxation curves for both short and long periods of time. For times greater than about 800 hr, a simpler approximate method of interpretation called the "intercept method" gives good agreement with the actual creep relaxation data.

In most creep tests, relations between creep deformation and time are obtained for essentially constant stress conditions. There are situations, however, where the stress in a member does not remain constant but relaxes or decreases with time. For example, in a bolted flange connection operating at an elevated temperature both the creep deformation in the flanges and the bolt produces a reduction or relaxation of the initial stress in the bolt. Stress relaxation may also occur in various products and parts made of plastics.

Tension relaxation tests are reported in this paper for both styrene and acrylic plastics. In these tests, a specimen is subjected first to a selected initial stress. Since the rate of loading was rapid, this is equivalent to loading to a selected initial strain. During the test, the strain for a given gage length is maintained constant. The total strain is made up of creep strain plus elastic strain. With time, the creep strain increases, so the elastic strain must have a corresponding decrease in order that there be a constant total strain. A decrease in the elastic strain must mean an accompanying reduction in stress. This reduction in stress with time is recorded in a relaxation test and stress-time relations as shown in Fig. 8 are obtained for various initial stress values.

Most creep tests are constant tensile stress tests in which the creep deformation is measured at constant stress for various intervals of time (Fig. 1). It seems desirable, therefore, to have available a theory or procedure for predicting

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* Presented at the Fifty-fourth Annual Meeting of the Society, June 18-22, 1951.

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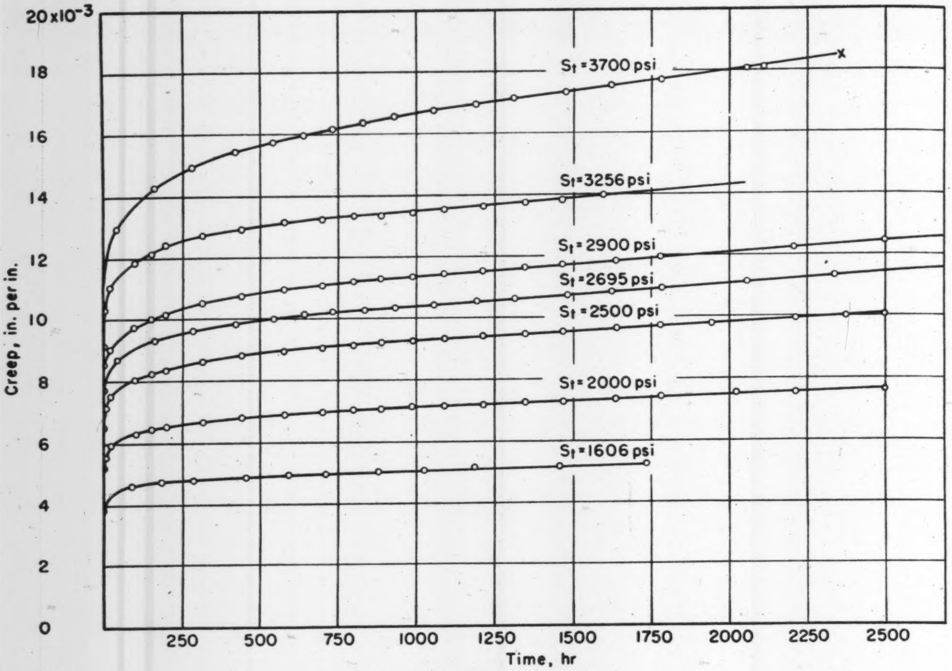
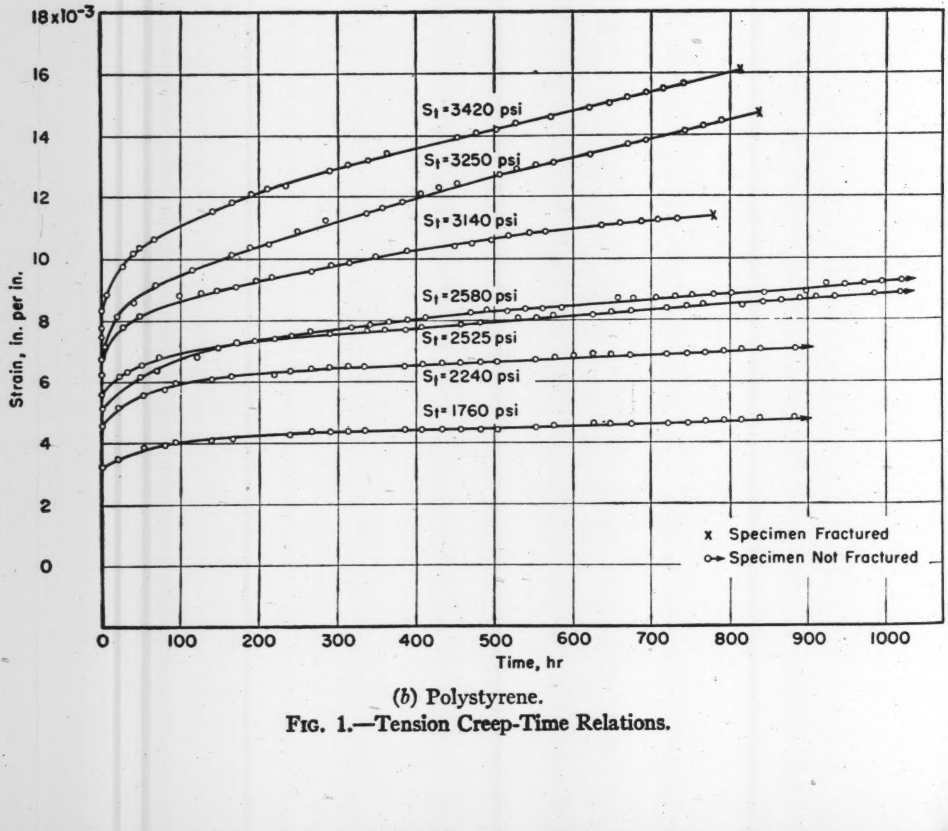


(*a*) Acrylic plastic.



(*b*) Polystyrene.

Fig. 1.—Tension Creep-Time Relations.

the stress-time relaxation relations based on the usual tension creep-time relations. One of the main objectives of this investigation was to develop theories for predicting stress-time relaxation relations from the constant stress creep-time test data. Several methods of interpretation were applied and two procedures were found to be suitable: a "strain-hardening method" (which agrees well with the actual creep relaxation relations for the entire time covered by the tests), and an "intercept method" which agrees with the test results for times other than the initial period, but which has the advantage that it is a simpler method than the "strain-hardening method." This "intercept method" is a limiting case of the "strain-hardening method."

TABLE I.—MECHANICAL PROPERTIES.

Material	Type of Stress	Modulus of Elasticity, psi	Fracture Strength, psi	Ductility at Fracture, per cent
Acrylic plastic....	Tension	4.3×10^{5}[a]	5000 to 7000[b]	1 to 15[b]
Styrene plastic....	Tension	4.5×10^{5}	5700[c]	1.35[c]

[a] Tension control test.

[b] From technical data supplied by Rohm & Haas Co., as determined by ASTM Standard Methods.

[c] J. A. Sauer, J. Marin and C. C. Hslao, "Creep and Damping Properties of Polystyrene," *Journal of Applied Physics*, Vol. 20, No. 6, pp. 505–517, June, 1949.

MATERIALS TESTED

The materials tested were Lustrex styrene plastic, manufactured by the Monsanto Chemical Co., and Plexiglas acrylic plastic, manufactured by Rohm & Haas Co. Values of the mechanical properties of these materials in tension are summarized in Table I.

The acrylic specimens were annealed after machining. This was done by rapid heating to 196 F and maintaining this temperature for 12 to 24 hr. The specimens were then slowly cooled to room temperature at a rate of about 10 F per hr. The SR-4 gages were then pasted on the specimens and the specimens were finally baked at 120 F for 24 hr. The styrene relaxation specimens were not annealed, since the constant stress creep specimens were tested in the unannealed condition.

All the creep tests were made in a creep laboratory in which the temperature was maintained at 77 ± 2 F, and the relative humidity was kept at 50 ± 2 per cent. A humidifier and a dehumidifier with automatic controls were used to maintain constant humidity. A constant temperature was maintained by use of heating coils with automatic controls.

CONSTANT CREEP STRESS TEST RESULTS

The usual constant stress creep tests in tension were made with types of specimens and equipment as described in an earlier paper (**1**)[2]. Deformation-time plots for various constant tensile stresses are shown in Fig. 1 for both the styrene and the acrylic plastics. The deformations plotted include both the elastic and creep deformations in keeping with usual practice. The creep-time relations shown are for as wide a range of stress values as possible.

INTERPRETATION OF TENSION CREEP-TIME DATA

There are a number of methods that can be used for the interpretation of the tension creep data (**2, 3, 4, 5**). These methods are of two kinds. That is, an empirical equation may be used to define the creep-time relation for a given stress and temperature or a relation between the creep, stress and time for a given temperature is defined. For the purpose of predicting stress relaxation relations based on the usual constant load tension creep test data, the creep-stress-time type of relation is necessary.

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[2] The boldface numbers in parentheses refer to the list of references appended tothis paper, see p. 1293.

In selecting a creep-stress-time expression to represent the tension creep behavior, it is necessary to select a relation with sufficient experimental constants so that a satisfactory fit of the test data is achieved. On the other hand, care must be exercised in avoiding the use of unnecessarily complicated expressions. In the course of this investigation, several creep-stress-time relations were analyzed. Two methods will be presented herein, designated as a "short-time relation" and a "long-time relation." The short-time relation gives a good fit with the creep-time curves for both the initial and long-time periods. On the other hand, the long-time relation is a simpler expression than the short-time relation, but it does not give a good fit to the test data during the initial period of time. For long periods of time, both methods are good approximations, and the simpler long-time relation is therefore recommended.

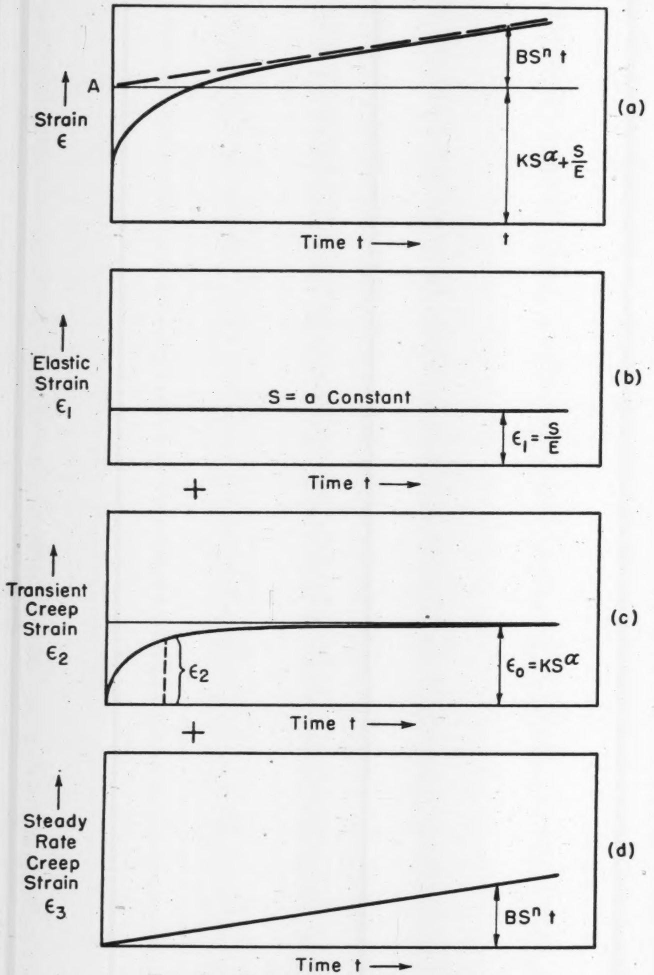

A
Strain
ϵ
$BS^n t$
$KS^{\alpha}+\frac{S}{E}$
(a)
Time t ⟶
t
Elastic
Strain
ϵ_1
S = a Constant
$\epsilon_1=\frac{S}{E}$
(b)
Time t ⟶
+
Transient
Creep
Strain
ϵ_2
ϵ_2
$\epsilon_0=KS^{\alpha}$
(c)
Time t ⟶
+
Steady
Rate
Creep
Strain
ϵ_3
$BS^n t$
(d)
Time t ⟶

FIG. 2.—Components of Creep Strain.

Short-Time Creep-Stress Relation:

By this method of interpretation the strain at a time t is considered to be made up of three parts (Fig. 2): (1) an elastic strain (ϵ_1), (2) a transient creep strain (ϵ_2), and (3) a steady rate creep strain (ϵ_3). That is, in Fig. 2(a), the total strain is,

$$\epsilon = \epsilon_1 + \epsilon_2 + \epsilon_3 \dots\dots\dots(a)$$

The values of the three strain components may be expressed analytically. The elastic strain is simply $\epsilon_1 = \frac{S}{E}$, where S is the stress and E is the modulus of elasticity. The transient creep strain reaches a limiting value as indicated in Fig. 2(c), since after an initial time period the creep rate becomes constant and all the creep is then represented by Fig. 2(d). The test data show that the limiting creep ϵ_0 (Fig. 2(c)), when plotted against the stress, gives a straight-line relation on a log-log plot. That is,

$$\log \epsilon_0 = \log K + \alpha \log S$$

or

$$\epsilon_0 = KS^{\alpha} \dots\dots\dots\dots\text{(b)}$$

where K and α are experimental constants. To provide for the initial variation between strain ϵ_2 and time t, a time function can be used and the creep ϵ_2 is then expressed as

$$\epsilon_2 = \epsilon_0(1 - Pe^{-pt}) = KS^{\alpha}(1 - Pe^{-pt}) \dots (c)$$

where:

P and p = experimental constants, and
e = the base for natural logarithms.

By Eq c, when t is large $\epsilon_2 \approx \epsilon_0 \approx KS^{\alpha}$. The steady rate creep strain represented in Fig. 2(d) can also be expressed analytically. It was found for the materials tested that there is a straight-line relation on a log-log plot between the creep rate and stress, that is,

$$\log C = \log B + n \log S$$

or

$$C = BS^n \dots\dots\dots\dots(d)$$

Noting that $\epsilon_3 = Ct$, Eq d can be written as

$$\epsilon_3 = BtS^n \dots\dots\dots\dots(e)$$

The total creep strain from Eqs c and e then becomes

$$\epsilon_c = \epsilon_2 + \epsilon_3$$

or

$$\epsilon_c = KS^{\alpha}(1 - Pe^{-pt}) + BS^nt \dots\dots(1)$$

The total strain is the sum of the creep strains and the elastic strain $\epsilon_1 = \frac{S}{E}$. That is, by Eqs a and 1

$$\epsilon = \epsilon_1 + \epsilon_2 + \epsilon_3 = \epsilon_1 + \epsilon_c$$

or

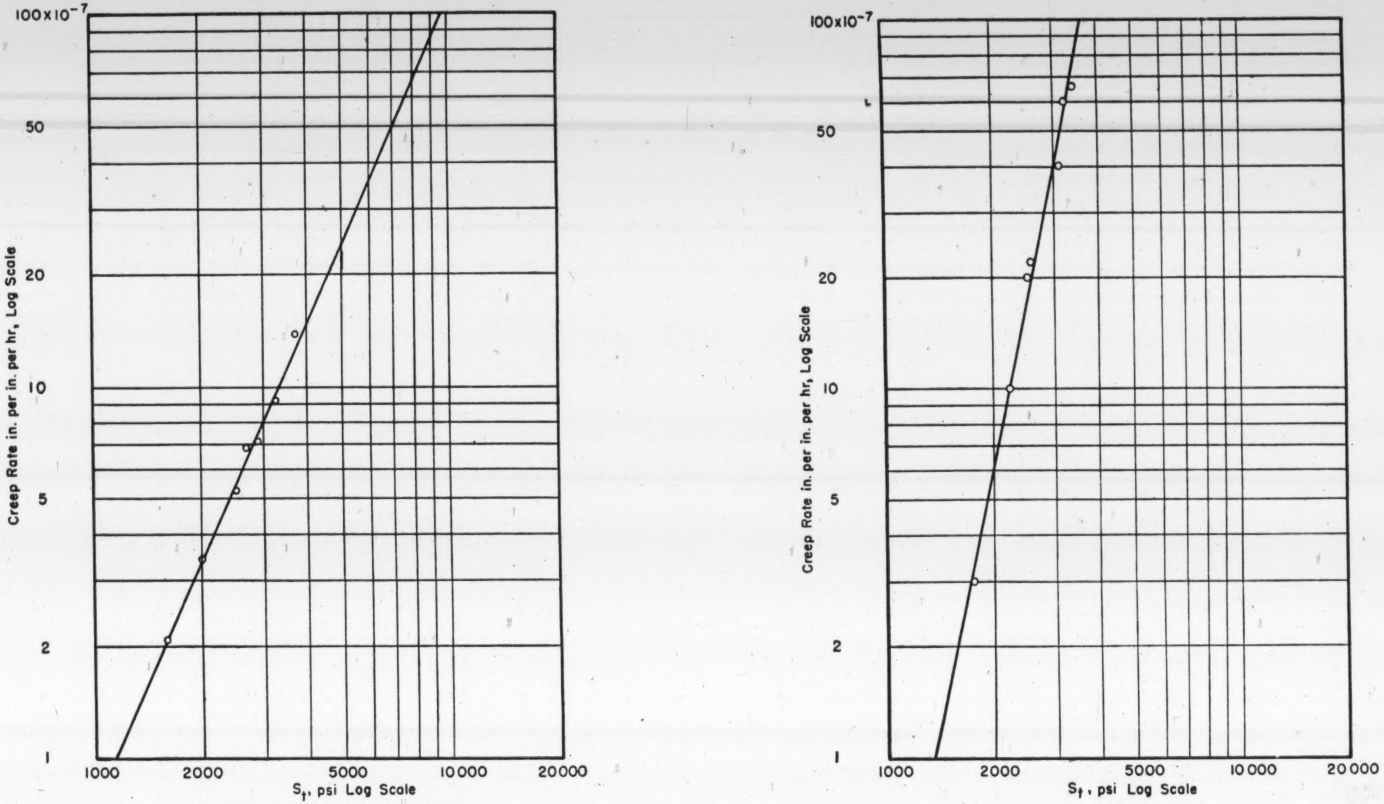
$$\epsilon = \frac{S}{E} + KS^{\alpha}(1 - Pe^{-pt}) + BS^nt \dots(2)$$

Equation 2 completely defines the total strain ϵ for a given stress S at a time t for the condition of constant stress. The experimental constants K, α, P, p, B and n in Eq 2 were obtained for each of the materials tested by the following procedure:

(1) From the creep-time relations of Fig. 1, the constant minimum creep rates, C, can be selected for the various stress values, S. A log-log plot between C and S shows a linear relation for both materials as indicated in Fig. 3. The best straight lines as shown were selected using the method of least squares. The constants B and n in the relation $C = BS^n$ obtained in this way are listed in Table II for both materials.

(2) The transient creep ϵ_2 can now be determined since

$$\epsilon_2 = \epsilon - (\epsilon_1 + \epsilon_3) = \epsilon - \frac{S}{E} - BtS^n. \quad (f)$$



(*a*) Acrylic plastic (annealed).

(*b*) Polystyrene.

FIG. 3.—Log-Log Creep Rate-Stress Relation for Tension.

That is, in Eq *f*, the total strain ϵ is given by the original creep-time relations of Fig. 1, the elastic strain $\frac{S}{E}$ is known for a given S stress since E can be determined, and the creep strain $\epsilon_3 = BS^nt$ is known since B and n are known. Values of the transient creep *versus* time as obtained by Eq *f* are shown plotted in Fig. 4 for both materials.

(3) From the plots of the transient creep in Fig. 4, the limiting creep strain values ϵ_0 can be selected for each stress value. These values are shown plotted against the log of the stress in Fig. 5 for each material. The method of least squares was used to obtain the best straight lines representing the test data. In this way, the experimental constants K and α in the relation $\epsilon_0 = KS^\alpha$ were obtained. For each material, Table II gives the values of the constants K and α.

TABLE II.—CONSTANT STRESS TENSION CREEP CONSTANTS.

For Acrylic Plastic (Annealed Material)

$$\epsilon \text{ total} = \frac{S}{E} + KS^\alpha (1 - P_e{}^{-pt}) + BS^nt$$

$E = 4.34 \times 10^5$	$p = 0.005$
$K = 1.00 \times 10^{-10}$	$B = 1.83 \times 10^{-14}$
$\alpha = 2.18$	$n = 2.19$
$P = 0.490$	

For Styrene Plastic

$$\epsilon \text{ total} = \frac{S}{E} + KS^\alpha (1 - P_e{}^{-pt}) + BS^nt$$

$E = 5.10 \times 10^5$	$p = 0.012$
$K = 3.11 \times 10^{-9}$	$B = 3.86 \times 10^{-22}$
$\alpha = 1.71$	$n = 4.60$
$P = 0.560$	

(4) With values of K and α known, the constants P and p in Eq *c* for the transient creep ϵ_2 can be determined. The values of P and p shown in Table II were determined by fitting Eq *c* to the transient creep curves and selecting an average value for the constants. When this was done, it was found that the transient creep-time relations for various stress values could be defined satisfactorily by Eq *c*.

By the foregoing procedure, the constants can be determined and the creep-time-stress relations can then be completely defined by Eq 2 for each material.

Long-Time Creep Stress Relation:

By the "long-time creep stress relation" no attempt is made to fit the empirical relation to the actual data during the initial time period. That is, the assumption is made that the creep strain is represented by

$$\epsilon_c = KS^\alpha + BS^nt \quad \ldots\ldots\ldots (3)$$

A comparison of Eqs 3 and 1 shows that the creep strain ϵ_c approximately agrees for large values of time, since the equations are identical when t becomes infinite.

Using Eq 3, the total strain can be written as

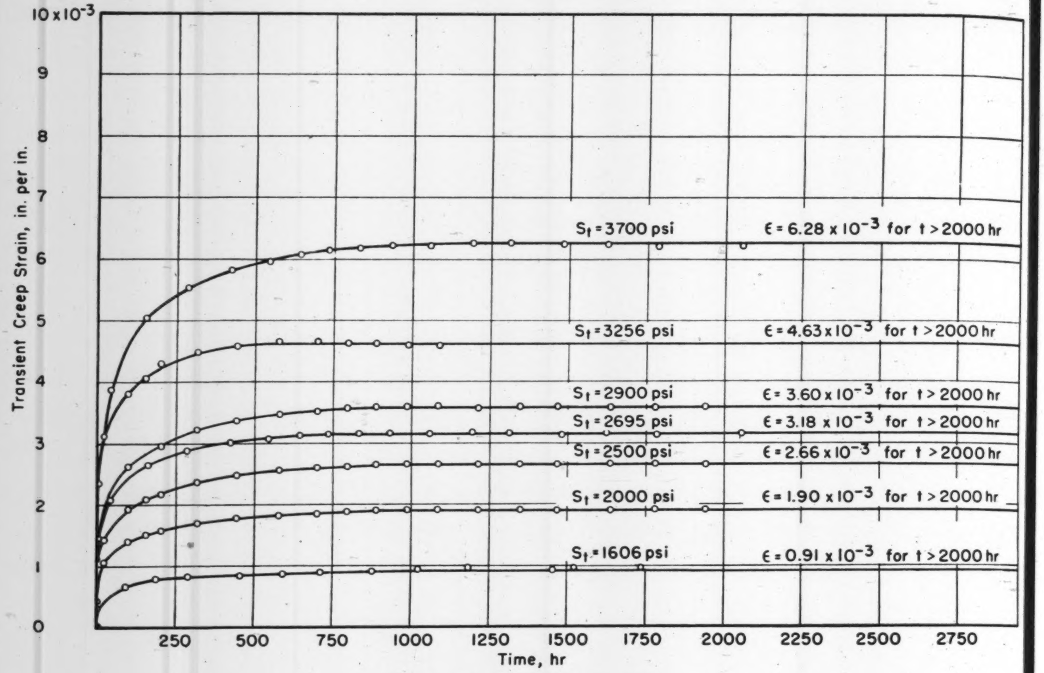
$$\epsilon = \epsilon_1 + \epsilon_c = \frac{S}{E} + KS^\alpha + BS^nt \quad .. \ (4)$$

The determination of the constants K, α, B and n in Eq 4 was explained in the discussion of the "short-time creep stress relation," and the values of these constants are given in Table II.

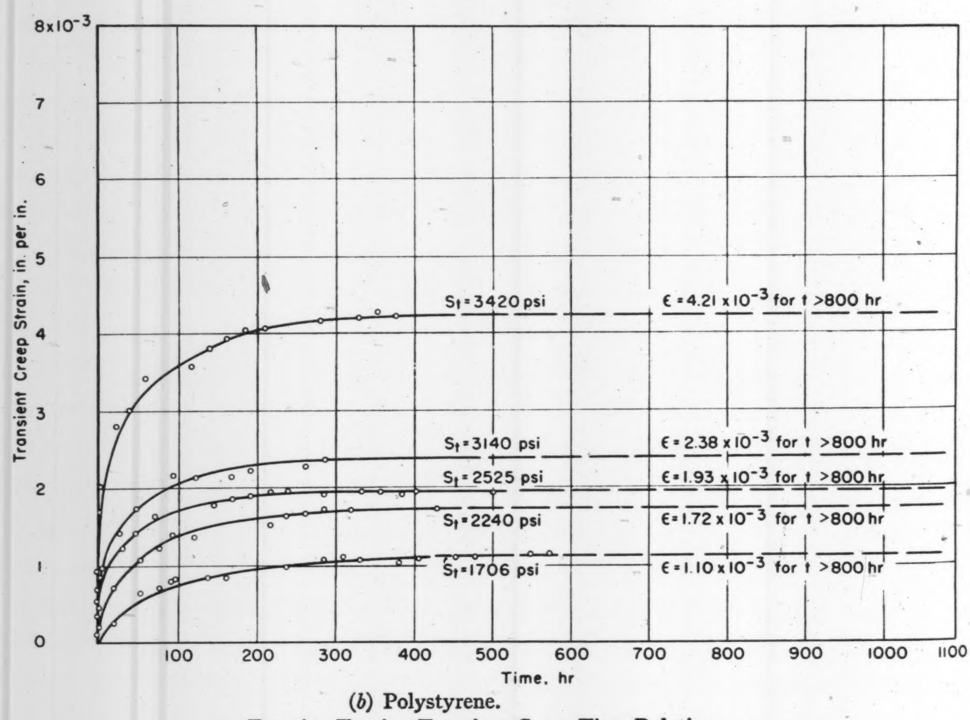
RELAXATION CREEP STRESS TEST RESULTS

The specimens used for the relaxation tension test results had dimensions as shown in Fig. 6. The specimens were tested in the specially designed 6-unit tension creep relaxation machine shown in Fig. 7.

The test procedure used consisted in loading a specimen, *Sp*, very rapidly to a predetermined total strain corresponding to a selected initial stress value. The total strain on the specimen is then kept constant by unloading the specimen gradually so that the increase in the creep strain is compensated for continuously by the decrease in the elastic strain produced by the decrease in the stress.

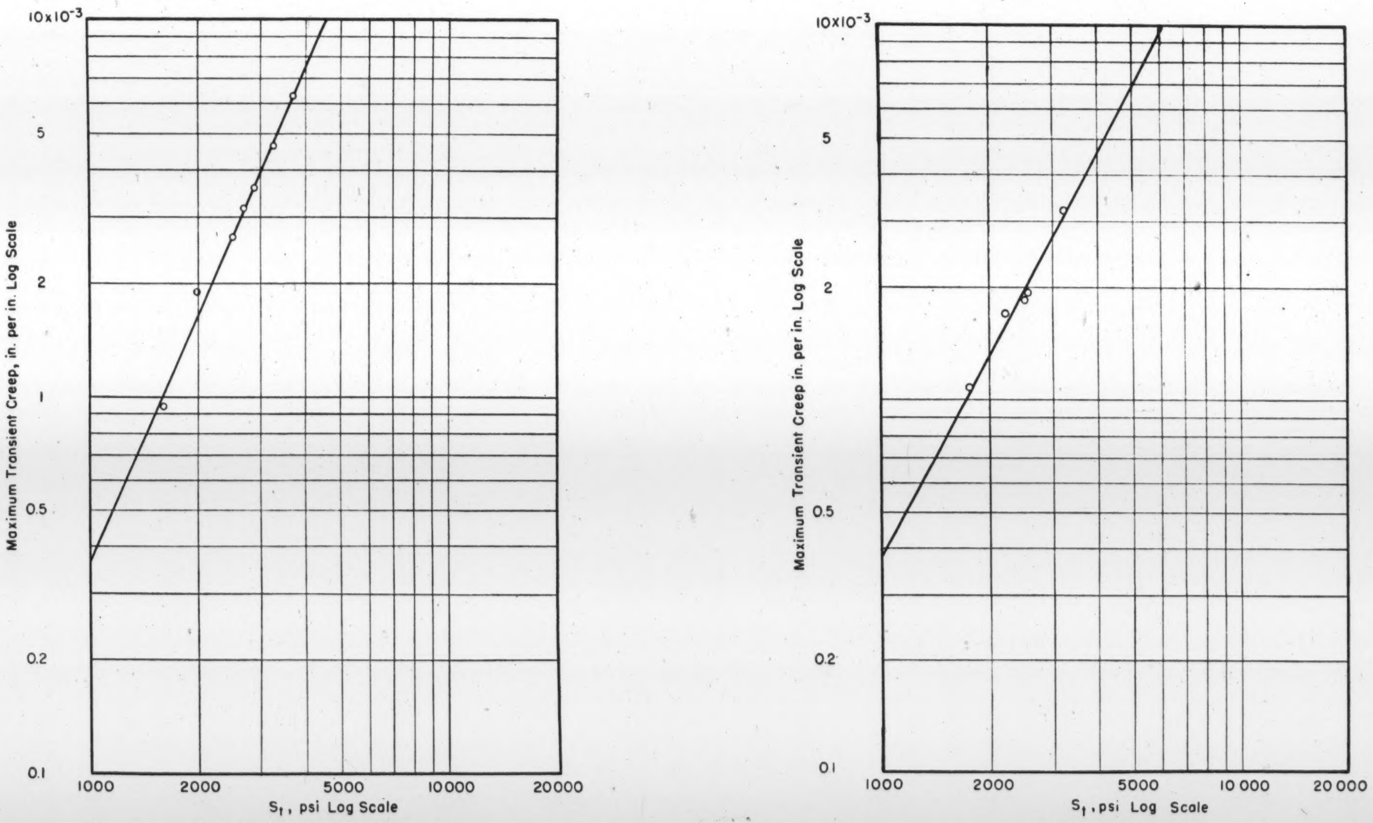


(*a*) Acrylic plastic (annealed).



(*b*) Polystyrene.

FIG. 4.—Tension Transient Creep-Time Relations.



(*a*) Acrylic plastic (annealed).

(*b*) **Polystyrene.**

FIG. 5.—Log-Log Maximum Transient Creep Stress Relations for Tension.

The tensile load is applied to the specimen by a variable speed reversible motor, *M*, at a rate of loading corresponding to 500 × 10^{-6} in. per in. per sec. Under this condition of rapid loading it can be assumed that the initial strain is entirely elastic. This assumption is confirmed by the fact that the values of the modulus of elasticity determined from the initial strains and stresses in the relaxation tests agree with values found in tension control tests. The deformations in the relaxation tests were measured by SR-4 electric strain gages with 13/16 in. gage lengths, *E*. The loads were measured by spring dynamometers, *D*. The dynamometers measure the loads by measuring the deflection of a helical spring using dial gages, *G*. The loads on the specimen were not reduced continuously but in small finite steps. At the beginning of each relaxation test, the unloading was done frequently, but after the initial stage, unloading was only required every day or so. At each unloading, values of the gage load reading and time were recorded for each specimen. With these data, the creep relaxation curves in Fig. 8 for each material were determined.

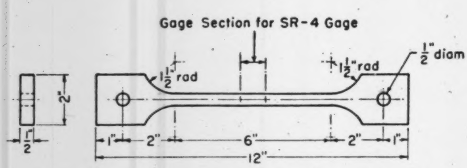


FIG. 6.—Dimensions of Specimens for Creep and Relaxation Tests.

DETERMINATION OF RELAXATION RELATIONS FROM CONSTANT STRESS CREEP TIME DATA

Most creep tests are conducted under constant tensile stress conditions. It would be desirable, therefore, if the creep relaxation relations for constant strain and decreasing stress could be predicted from the usual constant creep stress relations. Various theories and methods have been proposed for making such predictions (6, 7). This paper presents a "strain hardening method" which has been found to be in best agreement with the actual results. In applying this method, constant stress-creep-time relations given by Eqs 1 and 2 were used. A second approximate method called the "intercept method" is also given. This latter method is used with the "long-time creep-stress relation" represented by Eqs 3 and 4. For times other than the initial period, the "intercept method" is found to agree about as well with the actual creep relaxation values as the predictions by the "strain-hardening method."

Interpretation by "strain-hardening method":

The strain-hardening theory (6) assumes that the creep rate is only a function of the creep strain and stress. That is, irrespective of whether the strain is produced by creep or relaxation, there exists one creep rate for a given stress and creep strain, according to this theory. Referring to Fig. 9(*a*), for a given time t_1 and stress S_1 the strain is ϵ_{01} and the creep rate is the slope at point *A*. If the stress is now reduced or relaxed to a value S_2, it is of interest to know what the new creep rate will be. According to the strain-hardening theory, the creep rate will be equal to the slope at point *B* on the S_2 creep-time curve (Fig. 9(*a*)). On the other hand, according to the "time-hardening theory" (6), the creep rate for the new stress S_2 would be the slope at point *C* in Fig. 9(*a*). The time-hardening theory was found to give a poor prediction of the relaxation data herein obtained and will therefore not be discussed.

The creep strain-time relation corresponding to a reduction in stress from

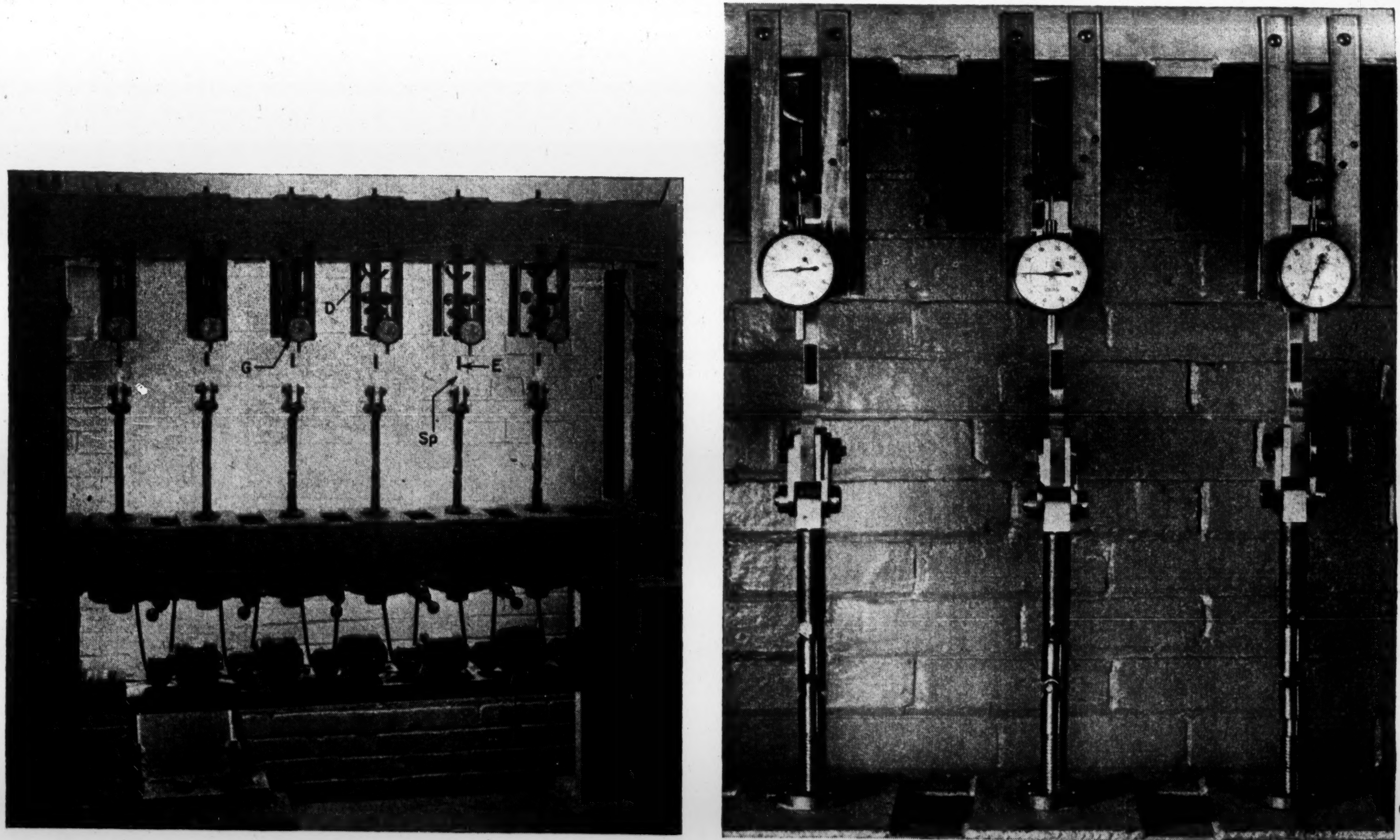
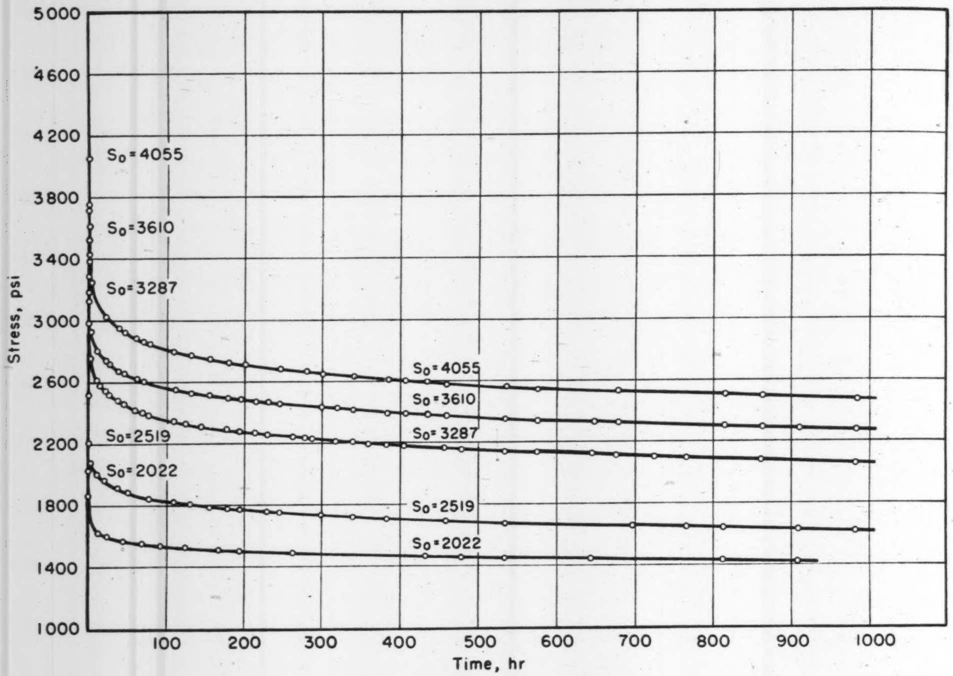
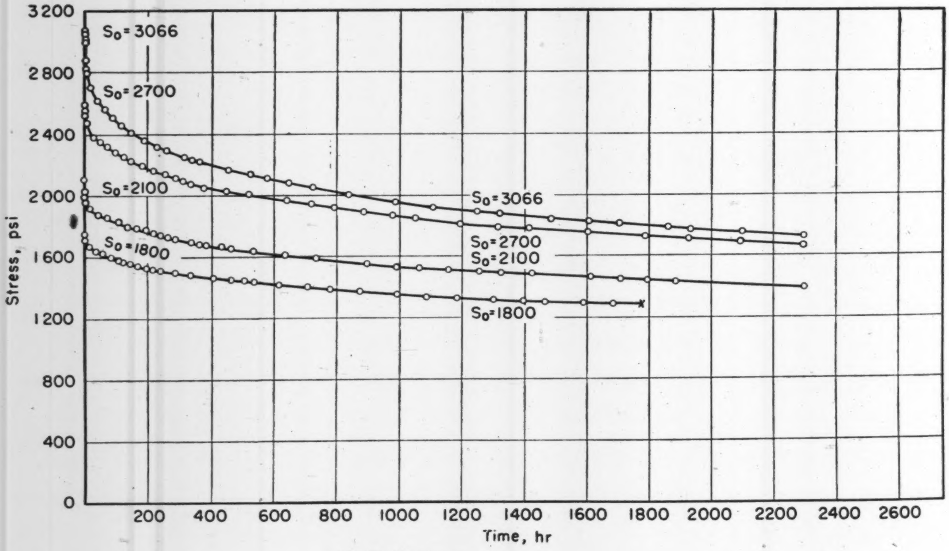


FIG. 7.—Stress Relaxation Testing Machine.



(*a*) Acrylic plastic (annealed).



(*b*) Polystyrene.

FIG. 8.—Relaxation of Tensile Stress.

S_1 to S_4 (Fig. 9(*a*)) is represented in Fig. 9(*b*). That is, the time corresponding to a relaxation of stress from S_1 to S_4 is

$$t = \Delta t_1 + \Delta t_2 + \Delta t_3 \ldots\ldots\ldots (g)$$

where Δt_1, Δt_2 and Δt_3 are shown in Fig. 9(*a*) and (*b*). If the time interval Δt is sufficiently small, Eq *g* can be written as

$$t = \Sigma \frac{\Delta\epsilon_c}{\left(\dfrac{\Delta\epsilon_c}{\Delta t}\right)} = \Sigma \frac{\Delta\epsilon_c}{C} = \int \frac{d\epsilon_c}{C}$$

That is

$$t = \int_0^{\epsilon_0} \frac{d\epsilon_c}{C} \ldots\ldots\ldots\ldots (h)$$

where C = the creep rate.

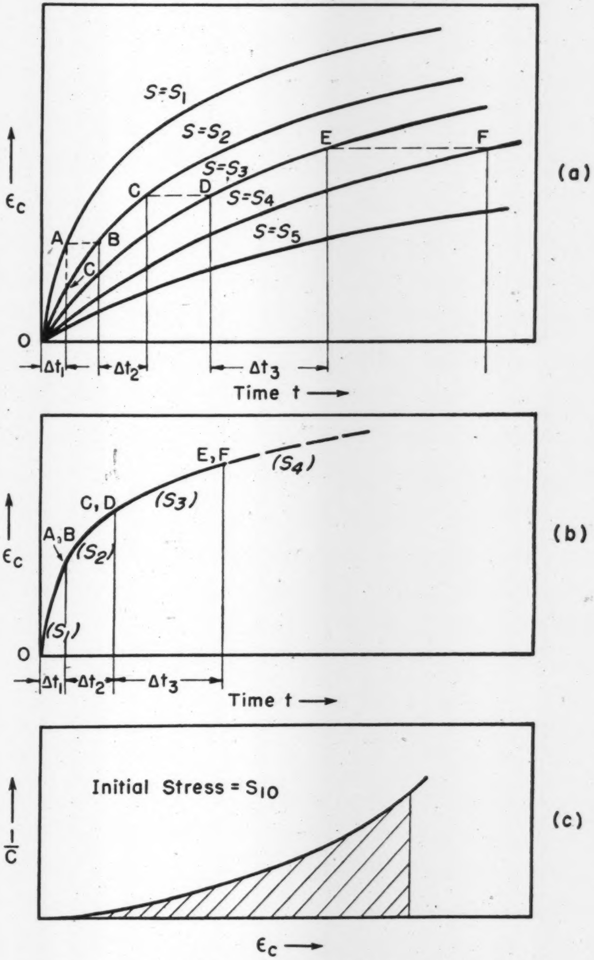
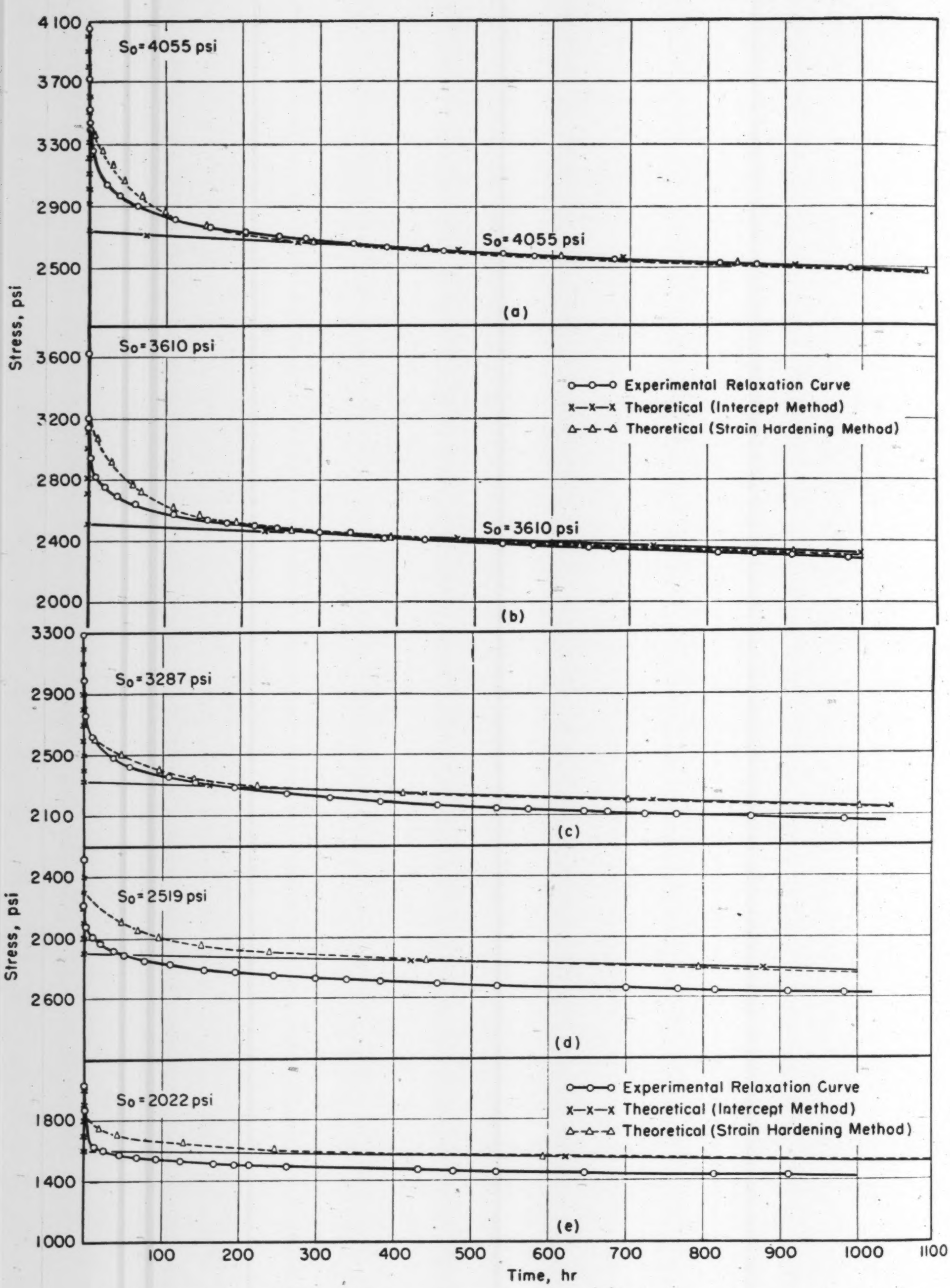


FIG. 9.—Illustration of Strain Hardening Method.

In a creep relaxation test, if S_0 is the initial stress and the stress is decreased to a value S, the decrease in the elastic strain $= -\left(\frac{S_0 - S}{E}\right)$. This decrease in the elastic strain equals the increase in the creep strain ϵ_c. That is, the upper



10.—Comparison of Theoretical and Actual Tension Relaxation Curves Acrylic Plastic (A d).

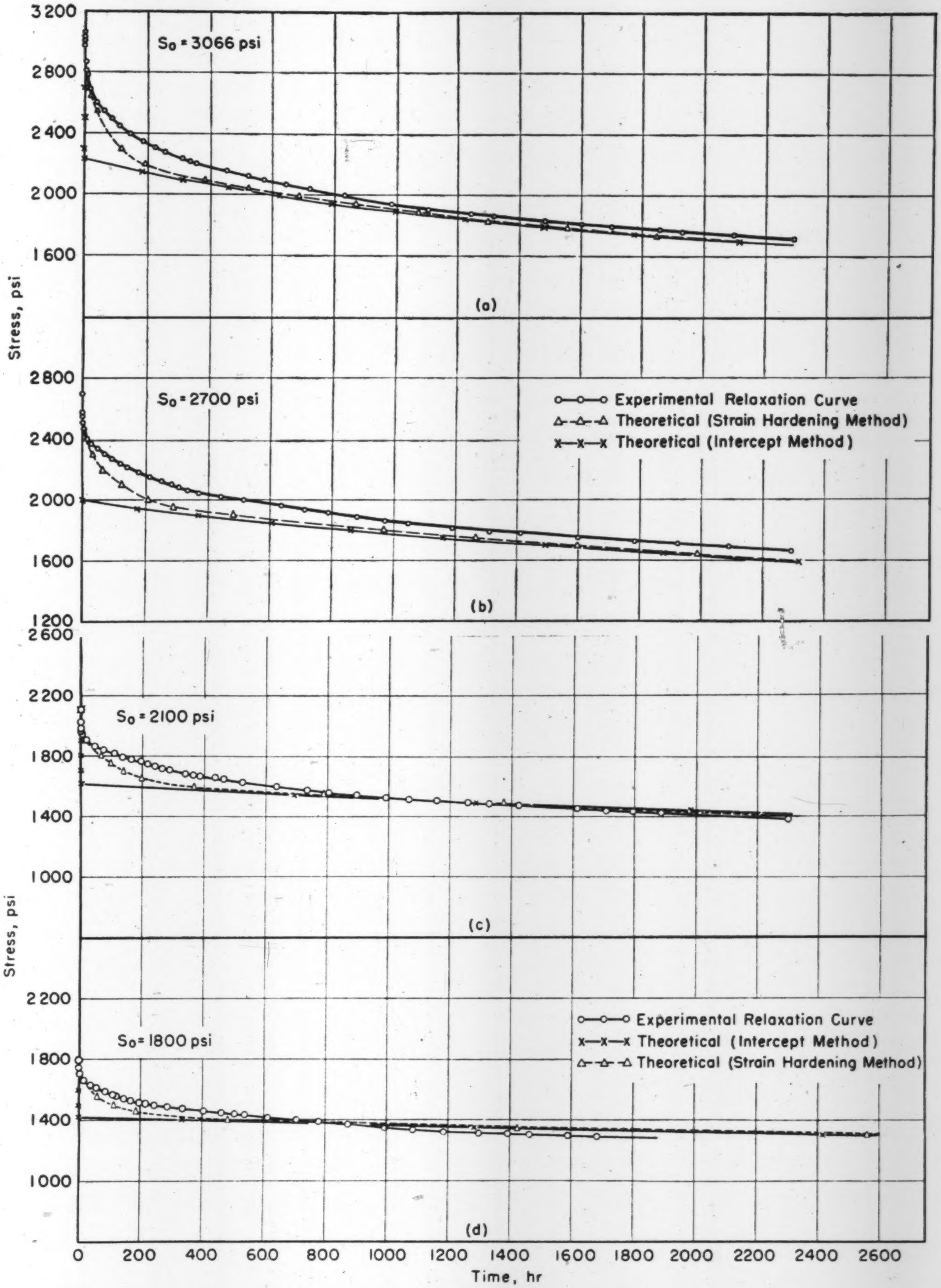


FIG. 11.—Comparison of Theoretical and Actual Tension Relaxation Curves Polystyrene.

limit ϵ_c in the integral of Eq *h* equals $\left(\frac{S_0-S}{E}\right)$. The evaluation of the integral in Eq *h* cannot be made directly since the creep rate cannot be conveniently expressed as a function of the stress and total creep strain using Eq 1. Under these circumstances, a graphical procedure can be used to evaluate the integral in Eq *h*. This procedure consisted of the following steps:

1. Using Eq 1 and values of creep constants obtained from the constant stress creep tests, a family of creep-time curves are constructed for selected values of the stress and for each material.

TABLE III(a).—PERCENTAGE DIFFERENCES BETWEEN PREDICTED AND ACTUAL STRESS RELAXATION VALUES FOR ACRYLIC PLASTIC (AT 1000 HR).

Specimen	Initial Stress S_0, psi	Experimental	Relaxed Stress, psi. Theorectical (Strain-Hardening)	Relaxed Stress, psi. Theoretical (Intercept Method)	Difference[a] (1), per cent	Difference[a] (2), per cent
No. 1	4055	2470	2470	2480	0	0.1
No. 2	3610	2265	2285	2295	1	1.4
No. 3	2519	1615	1765	1785	9.3	10.5
No. 4	3187	2055	2155	2155	4.9	4.9
No. 6	2022	1410	1520	1520	7.8	7.8

[a] Difference (1) Refers to difference between experimental values and theoretical values predicted by the strain hardening method.

Difference (2) Refers to difference between experimental values and theoretical values predicted by the intercept method.

2. For a decrease in stress from S_0 to S, the decrease in elastic strain is equal to the increase in creep strain which is $\epsilon_c = \frac{S_0-S}{E}$. For values of ϵ_c and S, the corresponding creep rates were determined from the creep curves constructed in 1.

3. A plot can then be made as shown in Fig. 9(*c*) giving the relation between $\frac{1}{C}$ and ϵ_c for a given initial stress S_0.

4. Calculations in (3) can be repeated for various values of the initial stress S_0.

5. From Fig. 9(*c*), the area under the curve is $\int \frac{d\epsilon_c}{C}$ and by Eq *h* this is the time *t* required to relax the stress from a value S_0 to S. By selecting a series of values of ϵ_c in Fig. 9(*c*), a series of values of time *t* and stress *S* corresponding to $\epsilon_c = \frac{S_0-S}{E}$ can be obtained. In this way, a theoretical stress-time relaxation relation is determined.

Figure 10 shows a comparison between the theoretical relaxation curves obtained in the foregoing manner with the actual relaxation curves. The percentage differences are given in Table III for the various initial stress values.

TABLE III(b).—PERCENTAGE DIFFERENCES BETWEEN PREDICTED AND ACTUAL STRESS RELAXATION VALUES FOR STYRENE PLASTIC (AT 1000 HR).

Specimen	Initial Stress S_0, psi	Experimental	Relaxed Stress, psi Theoretical (Strain-Hardening)	Relaxed Stress, psi Theoretical (Intercept Method)	Difference[a] (1), per cent	Difference[a] (2), per cent
No. 1	3066	1950	1920	1905	1.5	2.3
No. 2	2700	1865	1800	1780	3.5	4.5
No. 3	2100	1525	1520	1520	0.3	0.3
No. 6	2640	1345	1365	1360	1.9	1.8

[a] Difference (1) Refers to difference between experimental values and theoretical values predicted by the strain hardening method.

Difference (2) Refers to difference between experimental values and theoretical values predicted by the intercept method.

Interpretation by "intercept method":

By the intercept method, the "long-time creep relation" defined by Eq 3 will be used. That is, the creep-time relation in Fig. 2(*a*) is replaced by the two straight lines *OA* and *AB* so that the transient creep occurs suddenly along *OA*. In a relaxation test, the transient creep will also be assumed to occur suddenly so that the stress relaxes at once from a stress S_0 to S_1. With this assumption

$$\left(\frac{S_0 - S_1}{E}\right) = KS_1^{\alpha} \dots\dots\dots \quad (6)$$

That is, the sudden decrease in the elastic strain equals the sudden increase in the creep strain. The initial change in stress is followed by a gradual change in stress corresponding to the steady creep rate part of Eq 1. That is, the change in the elastic strain per unit of time equals the change in the creep strain per unit of time or

$$-\frac{d\epsilon_e}{dt} = \frac{d\epsilon_c}{dt} = C \quad \ldots\ldots\ldots\ldots (i)$$

The elastic strain $\epsilon_e = \dfrac{S_0 - S_1}{E}$ and by Eq 1, $C = BS^n$. Then Eq i can be written

$$-\frac{1}{E}\frac{dS}{dt} = BS^n \quad \ldots\ldots\ldots\ldots (j)$$

Integrating Eq (j):

$$t = \frac{1}{BE(n-1)}\left[S^{-(n-1)} - S_1^{-(n-1)}\right]. \quad (7)$$

Since B, n, E are known, by Eq 7, for a given stress S_1, the relation between the time t and stress S is defined. It is now possible for each initial stress value S_0 to first find the stress S_1 by Eq 6, and then by Eq 7 to determine the $S-t$ relations. These relations were obtained for various initial stress values and for both materials as shown in Fig. 10 and 11. A comparison of the stress-time relations as given by this "intercept method" with the actual values shows that except for the initial period the method gives a good approximation. The percentage differences between the values as given by this method and the actual values are shown in Table III.

CONCLUSIONS

1. Two methods of interpretation of constant stress creep test data are presented in this paper for acrylic and styrene plastics. One method, called the "short-time relation," gives a good approximation for both the initial period and for longer periods of time, while a "long-time relation," though simpler in form, is a good approximation only for the longer periods of time.

2. Two methods were determined for predicting the stress-time relaxation relations for tension. These theoretical relations were found to agree well with the actual relaxation curves.

Acknowledgments:

The authors acknowledge their appreciation to the Office of Naval Research of the U. S. Navy for the financial assistance and cooperation that made this project possible. They also wish to thank the Rohm & Haas Co. and The Monsanto Chemical Co. for supplying the materials tested.

REFERENCES

(1) J. Marin, "Static and Dynamic Creep Properties of Laminated Plastics for Various Types of Stress," Nat. Advisory Committee on Aeronautics *Tech. Note 1105*, Washington, D. C., Feb., 1947.

(2) J. Marin, "Mechanical Properties of Materials and Design," McGraw-Hill Book Co., New York, N. Y. (1942).

(3) A. H. Sully, "Metallic Creep," Interscience Publishers, Inc., 1949.

(4) E. G. Stanford, "The Creep of Metals and Alloys," Temple Press, Ltd., London (1949).

(5) G. V. Smith, "Properties of Metals at Elevated Temperatures," McGraw-Hill Book Co. (1950).

(6) C. C. Davenport, "Correlation of Creep and Relaxation Properties of Copper," *Journal of Applied Mechanics*, Vol. 5, No. 2, July, 1938, A55.

(7) C. R. Soderberg, "The Interpretation of Creep Tests for Machine Design," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 58, Nov., 1936, Paper RP 58-15, pp. 733–743.

DISCUSSION

Mr. George R. Gohn.[1]—Unfortunately, since the author's paper was not preprinted, I did not have an opportunity of reading it before today's meeting. However, I would like to ask a couple of questions relative to the work of the authors. As I understand it, SR-4 strain gages were used in these creep tests for measuring strain. Both the Canada Bureau of Mines and our own laboratories have used SR-4 gages for similar creep measurements. Our experimental work indicated that the creep of lead alloys as measured by SR-4 strain gages applied directly to the test specimen was always less than the extension measured by mechanical gages, and in some cases materially less. In one set of experiments where mechanical extensometers straddled SR-4 gages, the two sets of measurements agreed within 10 per cent for strain readings as great as 0.003. At greater values of strain the difference increased rapidly, but the SR-4 gage always indicated a substantially lower value of extension and a slower rate of creep than that indicated by the mechanical extensometer. In similar tests made at the Canada Bureau of Mines, the results differed in the order of 2 to 1; in other words, the SR-4 strain gage indicated only half the extension measured by the mechanical extensometer.

More recent tests using strain gages applied to one part of the test specimen and mechanical extensometers to another so that the two were in tandem showed even more marked differences. In other words, these tests indicated that SR-4 strain gages applied directly to a plastic material such as lead materially stiffened the gage length. I wonder whether the authors have observed similar phenomena in their tests on styrene and acrylic plastics. Such objections can of course be eliminated if the SR-4 gages were not mounted directly on the test specimens. I am also curious as to how the authors attached the strain gages to their test specimens, because we have had considerable difficulty in attaching gages to some of these materials by conventional methods.

Since the meeting, I have had an opportunity of reading the authors' paper and find that one section does not accurately define the authors' tests. For example, in the third paragraph of their paper the authors state that most creep tests are constant tensile stress tests in which the creep is measured at constant stresses for various intervals of time. They go on to discuss the constant stress-creep-time test, and in a subsequent section entitled "Constant Creep Stress Tests," give further indication that they have not accurately described either the bulk of the creep tests reported in the literature or their own tests. The tests which the authors have referred to are *constant load* tests; that is, tests in which a given load is applied to the test specimen to produce a given initial stress, and this load is allowed to remain on the test specimen either until failure occurs (that is the stress-rupture test), or until the test has been discontinued (in the long time creep test). A few investigators have made constant stress creep tests, but these tests require special equipment for removal of load in proportion to the increase in length of the test specimen. For example, McKeown in the September, 1950 issue of *Metallurgia,* Vol. 42, p. 189, describes a method for continually decreasing the load on the creep test specimen as the specimen elongates. Others have used different methods for accomplishing this, but I do not believe very many creep studies have been made at constant stress. Furthermore, all of the authors' tests are either of the constant load type or the constant strain type. In the latter, load is not decreased in proportion to the increase in strain, but rather decreased by an amount which will maintain a constant strain throughout the test.

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[1] Technical Staff, Bell Telephone Laboratories Inc., New York, N. Y.

MESSRS. JOSEPH MARIN AND YOH-HAN PAO (*authors' closure*).—The authors realize that in some cases the use of SR-4 gages is not suitable for creep strain measurements. However, these gages were found to be suitable for the creep strain measurements on plexiglas since the discrepancy between tension creep strain measurements at about 2000 hours using micrometer microscopes as compared to SR-4 gages was about 4 per cent. This was considered to justify the use of the SR-4 gages in creep tests of the materials reported in this paper. On the other hand, the authors found that SR-4 gages could not be attached satisfactorily to polystyrene for creep tests in compression. Apparently, inaccuracies due to poor bonding and restraining influence of the gages made the SR-4 gages unsuitable for use in tests of lead referred to by Mr. Gohn. However, this does not make these gages unsuitable for all materials and all sizes of specimens. The authors' check tests confirm this conclusion.

Mr. Gohn states that the constant stress creep tests referred to in the paper should be called constant load tests. Theoretically, he is correct, but actually the tests can be considered as constant stress tests since the variation in the stress in the tests reported, due to changes in cross-section, does not exceed 1.5 per cent—a negligible amount. Even if a constant stress machine were used, the error would still be about one-half of 1.5 per cent since corrections are not made for the elastic strains.

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON FLAME PHOTOMETRY

New testing methods using flame photometry were embodied in the Symposium on Flame Photometry presented at the sixth and ninth sessions of the Fifty-fourth Annual Meeting of the American Society for Testing Materials held in Atlantic City, N. J., on June 19, 1951. The Symposium was sponsored by Committee C-1 on Cement and Committee D-2 on Petroleum Products and Lubricants.

The following papers were presented:

Introduction—R. O. Clark
A Review of Flame Photometry—V. M. Meloche
Stable Internal Standard Flame Photometer for Sodium, Potassium, Lithium and Calcium Analyses in Biological Fluids and a Study of Ion Interference—C. L. Fox, Jr. and Elizabeth B. Freeman
Use of the Beckman and Perkin-Elmer Flame Photometers for the Determination of Alkalies in Portland Cement—J. J. Diamond and B. L. Bean
Applications of Flame Photometry to Materials Investigations—J. L. Gilliland
Determination of Lithium Oxide in Portland Cement—W. J. McCoy and G. G. Christiansen
Control of Interferences Caused by Acids and Salts in the Flame Photometric Determination of Sodium and Potassium—Frank T. Eggertsen, Garrard Wyld and Louis Lykken
The Effect of Organic Solvents on the Flame Photometric Emission of Certain Elements—G. W. Curtis, H. E. Knauer and L. E. Hunter
Determination of Tetraethyllead in Gasoline by Flame Photometry—P. T. Gilbert, Jr.
Determination of Calcium in Lubricating Oil by Flame Spectrophotometer—M. L. Moberg, V. B. Waithman, W. H. Ellis and H. D. DuBois
A Modified Recording Flame Photometer—W. H. King and W. M. Priestley
The Flame Photometer in the Analysis of Water and Water-Formed Deposits—R. K. Scott, V. M. Marcy and J. J. Hronas

These papers and a joint discussion have been issued by ASTM as Special Technical Publication No. 116 entitled, "Symposium on Flame Photometry."

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON STRUCTURAL SANDWICH CONSTRUCTIONS

This Symposium, held at the Twenty-third Session of the Fifty-fourth Annual Meeting of the American Society for Testing Materials in Atlantic City, N. J., June 21, 1951, under the sponsorship of Committee C-19 on Structural Sandwich Constructions, presented papers and discussions on structural sandwich constructions in order better to acquaint those interested with a wider knowledge of these constructions.

The following papers were presented:

Introduction—Albert G. H. Dietz
Developments and Trends in Lightweight Composite Construction—L. J. Markwardt
Sandwich Construction in the Elastic Range—H. W. March
Strength of Sandwich Construction—Charles B. Norris
Compressive and Torsional Instability of Sandwich Cylinders—George Gerard
Paper Honeycomb as a Core for Structural Sandwich Construction—Edward W. Kuenzi
Aluminum Honeycomb Sandwich Construction—Theodore P. Pajak
Some Developments in Structural Sandwich Building Panels Having Inorganic Cores—G. M. Rapp
Fabrication Techniques for Structural Sandwich Constructions—Bruce G. Heebink

These papers and discussions are issued as ASTM Special Technical Publication No. 118, entitled, "Symposium on Structural Sandwich Constructions."

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON BULK SAMPLING

Two sessions on Bulk Sampling were sponsored by Committee E-11 on Quality Control of Materials at the Fifty-fourth Annual Meeting of the American Society for Testing Materials held in Atlantic City, N. J., June 18, 1951. The papers "... report on the problems of sampling materials that occur in bulk form or in packages, with the aim of estimating, at minimum cost, measurable characteristics of a quantity of material in order to get some prescribed limit of error with an assigned probability."

The following papers are included:

Introduction—W. Edwards Deming
Materials Handling for Bulk Sampling—Joseph Manuele
Economic Accumulation of Variance Data in Connection with Bulk Sampling—Louis Tanner and Melvin Lerner
Two-Stage Acceptance Sampling by Attributes—C. W. Dunnett and J. W. Hopkins
Coal Sampling Problems—A. A. Orning
The Analysis of Variance in a Sampling Experiment—W. M. Bertholf
The Design of Coal Sampling Procedures—W. M. Bertholf

The papers together with discussions are issued as ASTM Special Technical Publication No. 114, entitled, "Symposium on Bulk Sampling."

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON MEASUREMENT OF CONSUMER WANTS

This Symposium was sponsored by the Administrative Committee on Ultimate Consumer Goods and covered soldier wants as well as researches in measuring consumer wants. It was presented at the Twentieth and Twenty-first sessions of the Fifty-fourth Annual Meeting of the American Society for Testing Materials held in Atlantic City, N. J., June 21, 1951.

The following papers were presented:

Determination of Soldier Wants—W. C. Schaefer
Determination of Soldier's Food Wants—R. Palmer Benedict
The General Problem of Measurement—Samuel A. Stouffer
Interviewer Bias—Clyde Hart
Some Application of the Panel Technique in Social Research—Charles Y. Glock
Effective Sampling Procedures—F. F. Stephan and P. J. McCarthy

These papers together with discussion have been issued by the ASTM as Special Technical Publication No. 117 entitled "Symposium on Measurement of Consumer Wants."

SUMMARY OF PROCEEDINGS OF THE CINCINNATI SPRING MEETING— SYMPOSIUM ON THERMAL INSULATING MATERIALS

The papers in this Symposium on Thermal Insulating Materials were presented at the 1951 Spring Meeting of the American Society for Testing Materials in Cincinnati, Ohio, March 7, 1951.

The Symposium was sponored by ASTM Technical Committee C-16 on Thermal Insulating Materials through a Special Symposium Committee headed by Mr. Ray Thomas, Staff Engineer, Carbide and Carbon Chemicals Co., Division of Union Carbide and Carbon Corp., South Charleston, W. Va. Mr. C. B. Bradley, Johns-Manville Corp., acted as Technical Chairman of the symposium session.

The following papers were presented:

- Introduction—Ray Thomas
- Basic Concepts of Water Vapor Migration and Their Application to Frame Walls—F. A. Joy
- Theory of, and Appropriate Methods for Measurement of Surface Emittance—L. P. Herrington
- Methods of Measurement and Application of the Theory of Specific Heat to Thermal Insulating Materials—Norman H. Spear
- Interlaboratory Comparison of Thermal Conductivity Determinations with Guarded Hot Plates—Henry E. Robinson and Thomas W. Watson
- Experiments with a Guarded Hot Plate Thermal Conductivity Set—C. F. Gilbo

These papers together with discussion are issued as ASTM Special Technical Publication No. 119 entitled, "Symposium on Thermal Insulating Materials."

Bertholf, W. M.
The Analysis of Variance in a Sampling Experiment—Summary of Proceedings of *STP 114*, 1298.
The Design of Coal Sampling Procedures—Summary of Proceedings of *STP 114*, 1298.

Bird, P. H.
Experience with Geophysics in New York State—Summary of Proceedings of *STP 122*, 1250.

Blank, A. E.
Discussion, 758.

Blank, A. I.
Discussion, 1020.

Blank, A. I., and Burghoff, H. L.
Creep Characteristics of Phosphorized Copper (0.019 per cent P) at 300, 400, and 500 F, 981. Disc., 994.

Blevins, T. B., and DeFries, M. G.
A Nomogram for Calculating the Stiffness of Elastomers. Published in ASTM BULLETIN, No. 173, April, p. 59 (TP113).

Bloem, D. L.
Crushing Tests of Sand, 339.
Effect of Certain Variables on Results of Standard Mortar Test of Fine Aggregates. Published in ASTM BULLETIN, No. 178, December, p. 44 (TP238).
See Walker, Stanton, and Bloem, Delmar L.

Boardman, H. C.
Discussion, 929.

Boor, Ladislav
See Landsberg, Meyer L., DiFilippo, Thomas J., and Boor, Ladislav.

Bouton, G. M.
Discussion, 743.

Bouton, G. M., and Phipps, G. S.
Compression Tests on Lead Alloys at Extrusion Temperatures, 761. Disc., 770.

Bowman, A. L.
See Helms, S. B., and Bowman, A. L.

Breen, John E.
Effect of Amplitude of Dynamic Stress on High Temperature Fatigue Life. Published in ASTM BULLETIN, No. 177, October, p. 38 (TP202).

Briggs, Thomas H., and Richard, C. D., Jr.
Development and Use of a D-C. Emission Figure of Merit for Oxide-Coated Cathodes, Published in ASTM BULLETIN, No. 171 January, p. 66 (TP28).

Brossman, M. W.
Discussion. Published in ASTM BULLETIN, No. 178, December, p. 50 (TP244).

Buffum, D. C.
See Jaffe, L. C., and Buffum, D. C.

Burghoff, H. L.
See Blank, A. I., and Burghoff, H. L.

Burmister, D. M.
The Application of Controlled Test Methods in Consolidation Testing—Summary of Proceedings of *STP 126*, 1249.

Byerly, F. S., Baumann, F. H., Diefenderfer, H. H., and Ashman, G. W.
A Laboratory Method of Test for No-Dirt-Retention Time of Traffic Paints. Published in ASTM BULLETIN, No. 176, September, p. 44 (TP182).

C

Cade, Arthur R.
An In-Vivo Method for Determining the Degerming Efficiency of Soaps Containing Hexachlorophene. Published in *STP 115*.

Cheesman, W. J.
Discussion, 1176.

Christiansen, G. G.
See McCoy, W. J., and Christiansen, G. G.

Clark, R. O.
Introduction to Symposium on Flame Photometry—Summary of Proceedings of *STP 116*, 1296.

Conrow, A. D.
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Cook, Herbert K.
Permeability Tests of Lean Mass Concrete, 1156.

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See Simmons, Ward F. and Cross, H. C.

Cross, H. C., and Van Echo, J. A.
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Cummings, J. D.
See Ellis, W. C., and Cummings, J. D.

Curtis, G. W., Knauer, H. E., and Hunter, L. E.
The Effect of Organic Solvents on the Flame Photometric Emission of Certain Elements—Summary of Proceedings of *STP 116*, 1296.

D

Dannenbrink, R. W.
See Fenske, M. R., Klaus, E. E., and Dannenbrink, R. W.

Davis, Evan A.
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Davis, R. E.
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Dawson, Raymond F.
See Matlock, L. H., and Dawson, R. F.
See Matlock, Hudson, Jr., Fenske, Carl W., and Dawson, Raymond F.

Deeming, W. Edwards
Introduction to Symposium on Bulk Sampling—Summary of Proceedings of *STP 114*, 1298.

DeFries, M. G.
See Blevins, T. B., and DeFries, M. G.

Demer, L. J.
See Lazan, B. J., and Demer, L. J.

Diamond, J. J., and Bean, B. L.
Use of the Beckman and Perkin-Elmer Flame Photometers for the Determination of Alkalies in Portland Cement—Summary of Proceedings of *STP 116*, 1296.

Diefenderfer, H. H.
See Byerly, F. S., Baumann, F. H., Diefenderfer, H. H., and Ashman, G. W.

Dietz, Albert G. H.
Introduction to Symposium on Structural Sandwich Constructions—Summary of Proceedings of *STP 118*, 1297.

DiFilippo, Thomas J.
See Landsberg, Meyer I., DiFilippo, Thomas J., and Boor, Ladislav.

Dolan, Thomas J.
Discussion, 608, 647, 719, 758, 853, 1085.
Electrically Excited Resonant-Type Fatigue Testing Equipment. Published in ASTM BULLETIN, No. 175, July, p. 60 (TP150). Disc., p. 67 (TP157).

Dollins, C. W.
Discussion, 743.

Dorn, J. E.
See Sherby, O. D., and Dorn, J. E.
See Sherby, O. D., Tietz, T. E., and Dorn, J. E.

Dowling, J. D.
See Abdun-Nur, E. A., and Dowling, J. D.

Draves, Carl Z.
Draves Wetting Test Method. Published in *STP 115*.

DuBois, H. D.
See Moberg, M. L., Waithman, V. B., Ellis, W. H., and DuBois, H. D.

Dulis, E. J.
Discussion, 994.
See Smith, G. V., Dulis, E. J., and Houston, E. G.

Dunn, E. J., Jr.
Film Thickness Measurements. Published in ASTM BULLETIN, No. 172, February, p. 35 (TP49).

Dunnett, C. W., and Hopkins, J. W.
Two-Stage Acceptance Sampling by Attributes—Summary of Proceedings of *STP 114*, 1298.

E

Eash, J. T., and Gagnebin, A. P.
Arbitration Bar Izod Impact Test for Cast Iron, 1061. Disc., 1070.

Eckel, E. B.
Interpreting Geologic Maps for Engineers—Summary of Proceedings of *STP 122*, 1250.

Eckel, John F.
Influence of Frequency on Repeated Bending Life of Acid Lead, 745. Discussion, 759.
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Eggertsen, Frank T., Wyld, Garrard, and Lykken, Louis
Control of Interferences Caused by Acids and Salts in the Flame Photometric Determination of Sodium and Potassium—Summary of Proceedings of *STP 116*, 1296.

Elling, R. E.
See Newmark, N. M., Mosborg, R. J., Munse, W. H., and Elling, R. E.

Ellis, W. C.
See Gohn, G. R., and Ellis, W. C.
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Ellis, W. C., and Cummings, J. D.
Creep Test Methods for Determining Cracking Sensitivity of Polyethylene Polymers. Published in ASTM BULLETIN, No. 177, December, p. 47 (TP241). Disc., p. 50 (TP 244).

Ellis, W. H.
See Moberg, M. L., Waithman, V. B., Ellis, W. H., and DuBois, H. D.

Emerson, R. W.
Fabrication of Chromium-Molybdenum Steel Piping for 1000 to 1500 F Central Station Service, 869. Disc., 893.

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F

Fenske, Carl W.
See Matlock, Hudson, Jr., Fenske, Carl W., and Dawson, Raymond F.

Fenske, M. R., Klaus, E. E., and Dannenbrink, R. W.
Viscosity—Shear Behavior of Two Non-Newtonian Polymer-Blended Oils, Symposium on Methods of Measuring Viscosity at High Rates of Shear. Published in *STP 111*.

Fessler, William A.
Measurement of the Adsorption of Anion-Active Detergents by Materials Commonly Washed. Published in *STP 115*.

Finley, G. S., Morrison, H. L., and Ragan, H. S.
A Test of a Prestressed Concrete Beam. Published in ASTM BULLETIN, No. 177, October, p. 61 (TP225).

Finn, F. N.
The Effect of Temperature on the Consolidation Characteristics of Remolded Clay—Summary of Proceedings of *STP 126*, 1249.

Fischer, H. C.
See Murray, J. A., and Fischer, H. C.

Fluck, P. G.
Influence of Surface Roughness on Fatigue Life and Scatter of Test Results of Two Steels, 584. Disc., 592.

Fox, C. L., Jr., and Freeman, Elizabeth B.
Stable Internal Standard Flame Photometer for Sodium, Potassium, Lithium and Calcium Analyses in Biological Fluids and a Study of Ion Interference—Summary of Proceedings of *STP 116*, 1296.

Frankel, L. P., and Radcliffe, C. W.
Bearing Strength of Laminated Plastics. Published in ASTM BULLETIN, No. 175, July, p. 71 (TP161).

Freeman, Elizabeth B.
See Fox, C. L., Jr., and Freeman, Elizabeth B.

Freudenthal, Alfred M.
Planning and Interpretation of Fatigue Tests—Summary of Proceedings of *STP 121*, 583. Discussion, 806.

Frisch, David H., and Levin, Jules S.
A Mechanical Wear Test Using Fission Fragments. Published in ASTM BULLETIN, No. 172, February, p. 64 (TP78).

G

Gagnebin, A. P.
See Eash, J. T., and Gagnebin, A. P.

Gardner, F. S.
Discussion, 977.

Geniesse, J. C.
Introduction to Symposium on Methods of Measuring Viscosity at High Rates of Shear, Symposium on Methods of Measuring Viscosity at High Rates of Shear. Published in *STP 111*.

Gerard, George.
Compressive and Torsional Instability of Sandwich Cylinders—Summary of Proceedings of *STP 118*, 1297.

Gibbs, H. J.
See Holtz, W. G. and Gibbs, H. J.

Gilbert, P. T., Jr.
Determination of Tetraethyllead in Gasoline by Flame Photometry—Summary of Proceedings of *STP 116*, 1296.

Gilbo, C. F.
Experiments with a Guarded Hot Plate Thermal Conductivity Set—Summary of Proceedings of *STP 119*, 1300.

Gilliland, J. L.
Applications of Flame Photometry to Materials Investigations—Summary of Proceedings of *STP 116*, 1296.

Girton, W. Z., and Potter, J. H.
Apparatus and Technique for Measuring Thermal Conductivity of Powdered Material. Published in ASTM BULLETIN, No. 172, February, p. 47 (TP61).

Glock, Charles Y.
Some Application of the Panel Technique in Social Research—Summary of Proceedings of *STP 117*, 1299.

Gohn, G. R.
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Gohn, G. R., and Ellis, W. C.
Fatigue Test as Applied to Lead Cable Sheath, 721. Disc., 743.

Goldspiel, S., and Bernstein, F.
Application of X-ray Diffraction Methods to the Identification of Natural and Synthetic Rubbers. Published in ASTM BULLETIN, No. 171, January, p. 71 (TP33).

Gonnerman, H. F. and Lerch, William
Changes in Characteristics of Portland Cement as Exhibited by Laboratory Tests over the Period 1904 to 1950. Published as *STP 127*.

Gordon, S. A.
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Greenman, R. L.
The Engineer Looks at Pedology—Summary of Proceedings of *STP 122*, 1250.

Grinnell, W. W.
See Williams, J. L., and Grinnell, W. W.

Gross, M. R.
Discussion. Published in ASTM BULLETIN, No. 175, July, p. 67 (TP157).
Laboratory Evaluation of Materials for Marine Propulsion Gears, 701. Disc., 718.

Gruntfest, I. J.
Mechanism of the Wetting of Textiles. Published in *STP 115*.

Guins, S. G., and Kell, J. A.
Vibrations in Railroad Freight Cars. Published in ASTM BULLETIN, No. 173, April, p. 46 (TP100).

H

Hand, W., and Winans, R. R.
An Apparatus for Measuring Dielectric Strength at Elevated Temperatures. Published in ASTM Bulletin, No. 171, January, p. 63 (TP25).

Harris, J. C.
Introduction to Papers on Evaluation of Soaps and Detergents. Published in *STP 115*.

Hart, Clyde
Interviewer Bias—Summary of Proceedings of *STP 117*, 1299.

Hastings, Carlton H.
An Elevation of Radiography with Particular Emphasis on Detection Methods. Published in ASTM Bulletin, No. 172, February, p. 66 (TP80).

Hebeler, H. H., Kolb, H. J., Stillman, J. W., and Baldt, J. H.
An Improved Electronic Flexometer for Bending Analysis and Stiffness Studies of Fabrics and Thin Plastics. Published in ASTM Bulletin, No. 176, September, p. 52 (TP190).

Heebink, Bruce G.
Fabrication Techniques for Structural Sandwich Constructions—Summary of Proceedings of *STP 118*, 1297.

Heller, J. J.
Discussion, 912.

Helms, S. B.
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Method of Making Vibrated Dry-Tamp Concrete Cylinders Applied to Tests of Lightweight Aggregate and Block Mixtures, 1184. Disc., 1196.

Hensley, J. W., Skinner, H. A., and Suter, H. R.
A Metal Cleaning Test Using Radioactive Stearic Acid as Soil. Published in *STP 115*.

Herrington, L. P.
Theory of, and Appropriate Methods for Measurement of Surface Emitance—Summary of Proceedings of *STP 119*, 1300.

Hodge, J. M.
See Rickett, R. L., and Hodge, J. M.

Holler, Albert C.
Sampling of Ferrous and Non-Ferrous Alloys—A Bibliography. Published in ASTM Bulletin, No. 174, May, p. 66 (TP136).

Holmes, B. M.
An Electric Timing and Operating Mechanism for a Bitumen Testing Penetrometer. Published in ASTM Bulletin, No. 175, July, p. 81 (TP171).

Holtz, W. G., and Gibbs, H. J.
Consolidation and Related Properties of Loess Soils—Summary of Proceedings of *STP 126*, 1249.

Hopkins, J. W.
See Dunnett, C. W. and Hopkins, J. W.

Horger, O. J.
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Hough, B. K.
An Analysis of the Effect of Particle Interlocking on the Strength of Cohesionless Soil. Published in ASTM Bulletin, No. 176, September, p. 55 (TP193).

Houston, E. G.
See Smith, G. V., Dulis, E. J., and Houston, E. G.

Howard, F. L.
See Mallonee, R. E., and Howard, F. L.

Howell, F. M.
See Aber, W. C., and Howell, F. M.
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Hronas, J. J.
See Scott, R. K., Marcy, V. M., and Hronas, J. J.

Hsiao, C. C., and Sauer, J. A.
Effect of Strain Rate on the Tensile and Compressive Stress-Strain Properties of Polystyrene. Published in ASTM Bulletin, No. 172, February, p. 29 (TP43).

Hu, L. W.
See Marin, Joseph, and Hu, L. W.

Hunter, L. E.
See Curtis, G. W., Knauer, H. E., and Hunter, L. E.

J

Jack, William
Basic Physical Properties of Acoustical Materials—Summary of Proceedings of *STP 123*, 1248.

Jacobson, M. M.
Discussion, 1059.

Jaffe, L. D., and Buffum, D. C.
Reproducibility of Bend Test for High Hardness Steels. Published in ASTM Bulletin, No. 177, October, p. 36 (TP200).

Jensen, W. L.
See Seens, W. B., Jensen, W. L., and Miller, O. O.

Johnson, A. M., and Wesley, R. H.
Application of Seismic Methods to Foundation Exploration—Summary of Proceedings of *STP 122*, 1250.

Joy, F. A.
Basic Concepts of Water Vapor Migration and Their Application to Frame Walls—Summary of Proceedings of *STP 119*, 1300.

K

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A Rapid Technique of Consolidation Testing—Summary of Proceedings of *STP 126*, 1249.

Keeley, Armand E.
Roundness Testing of Glass Spheres. Published in ASTM BULLETIN, No. 174, May, p. 72 (TP 142).

Kell, J. A.
See Guins, S. G., and Kell, J. A.

King, Myrle E.
See Mielenz, Richard C., and King, Myrle E.

King, W. H., and Priestley, W. M.
A Modified Recording Flame Photometer—Summary of Proceedings of *STP 116*, 1296.

Kinnunen, Jorma
Discussion. Published in ASTM BULLETIN, No. 177, October, p. 39 (TP203).

Kinsey, H. V.
An Evaluation of the Effective Gage-Length Equivalent of the Fillet and Shoulder of the Gage Length Portion of a Tension Test Bar Under Creep and Stress-Rupture Conditions. Published in ASTM BULLETIN, No. 171, January, p. 60 (TP22).

Klaus, E. E.
See Fenske, M. R., Klaus, E. E., and Dannenbrink, R. W.

Kleppinger, D. H.
See Babington, W. C., and Kleppinger, D. H.

Klima, Stanley J.
See Marchese, Vincent J., and Klima, Stanley J.

Kline, G. M.
Discussion. Published in ASTM BULLETIN, No. 178, December, p. 50 (TP244).

Knauer, H. E.
See Curtis, G. W., Knauer, H. E., and Hunter, L. E.

Kolb, H. J.
See Hebeler, H. H., Kolb, H. J., Stillman, J. W., and Baldt, J. H.

Kretsinger, D. G.
See Price, W. H., and Kretsinger, D. G.

Krivobok, V. N.
See Muhlenbruch, C. W., Krivobok, V. N., and Mayne, C. R.

Kuenzi, Edward W.
Paper Honeycomb as a Core for Structural Sandwich Construction—Summary of Proceedings of *STP 118*, 1297.

L

Landsberg, Meyer I., DiFilippo, Thomas J., and Boor, Ladislav
Properties of Exposed and Unexposed Polyvinyl Butyral Coated Fabrics, 1262. Disc., 1276.

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Damping, Elasticity, and Fatigue Properties of Temperature-Resistant Materials, 611. Disc., 647.

Lazan, B. J., and Wu, T.
Damping, Fatigue, and Dynamic Stress-Strain Properties of Mild Steel, 639. (*R*) Disc., 679.

Leedy, H. A.
Activity of Committee C-20—Summary of Proceedings of *STP 123*, 1248.

Leonard, E. A.
Laboratory Performance Test for Detergents in Continuous Wool Scouring. Published in *STP 115*.

Lerch, William
See Gonnerman, H. F. and Lerch, William.

Lerner, Melvin
See Tanner, Louis and Lerner, Melvin.

Lessells, J. M.
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Levin, Jules S.
See Frisch, David H., and Levin, Jules S.

Linehan, D.
Seismology Applied to Shallow Zone Research—Summary of Proceedings of *STP 122*, 1250.

Link, H. S.
See Smith, G. V., Seens, W. B., Link, H. S., and Malenock, P. R.

Litehiser, R. R.
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Loewer, A. C., Eney, W. J., Seymour, R., and Pascoe, W.
Physical Properties of Plasticized Sulfur Cements, 1234.

Lord, G. W., and Willis, T. F.
Calculation of Air Bubble Size Distribution from Results of a Rosiwal Traverse of Aerated Concrete. Published in ASTM BULLETIN, No. 177, October, p. 56 (TP220).

Lueder, D. R.
The Preparation of an Engineering Soil Map of New Jersey—Summary of Proceedings of *STP 122*, 1250.

Lykken, Louis
See Eggertsen, Frank T., Wyld, Garrard, and Lykken, Louis.

M

MacKenzie, J. T.
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McCarthy, P. J.
See Stephan, F. F., and McCarthy, P. J.

McCoy, W. J., and Christiansen, G. G.
Determination of Lithium Oxide in Portland Cement—Summary of Proceedings of *STP 116*, 1296.

McFadden, Gayle, and Ricketts, Walter C.
Corps of Engineers' Method for Design and Control of Asphalt Paving Mixtures. Published in ASTM BULLETIN, No. 171, January, p. 39 (TP1).

Machlowitz, Roy A.
See Pfleiderer, Howard G., and Machlowitz, Roy A.

Malenock, P. R.
See Smith, G. V., Seens, W. B., Link, H. S., and Malenock, P. R.

Mallonee, R. E., and Howard, F. L.
The Determination of Freezing Point of Engine Antifreeze. Published in ASTM BULLETIN, No. 172, February, p. 43 (TP57).

Manjoine, M. J.
Discussion, 979, 980, 1084.

Mansur, C. I.
See Shockley, W. G., and Mansur, C. I.

Manuele, Joseph
Materials Handling for Bulk Sampling—Summary of Proceedings of *STP 114*, 1298.

March, H. W.
Sandwich Construction in the Elastic Range —Summary of Proceedings of *STP 118*, 1297.

Marchese, Vincent J., and Klima, Stanley J.
A Test Method for Determining Fogging Properties of Organic Materials. Published in ASTM BULLETIN, No. 172, February, p. 40 (TP54).

Marcy, V. M.
See Scott, R. K., Marcy, V. M., and Hronas, J. J.

Marin, Joseph, and Hu, L. W.
Interpretation of Tension Creep-Time Relations. Published in ASTM BULLETIN, No. 171, January, p. 57 (TP19).

Marin, Joseph, and Pao, Yoh-Han
Creep Relaxation Relations for Styrene and Acrylic Plastics, 1277. Disc., 1295.

Markwardt, L. J.
An Idea Whose Time Had Come—Annual Address by the President, 27.
Developments and Trends in Lightweight Composite Construction—Summary of Proceedings of *STP 118*, 1297.
Highlights of Progress in Forest Products Research. Published in ASTM BULLETIN, No. 174, May, p. 45 (TP115).

Markwood, W. H., Jr.
See Woodham, Howard W., Wirick, Max G., and Markwood, W. H., Jr.

Mather, Bryant
Tests of Fine Aggregate for Organic Impurities and Compressive Strength in Mortars. Published in ASTM BULLETIN, No. 178, December, p. 35 (TP229).
Discussion, 1135, 1183.

Matlock, Hudson, Jr., Fenske, Carl W., and Dawson, Raymond F.
De-aired, Extruded Soil Specimens for Research and for Evaluation of Test Procedures. Published in ASTM BULLETIN, No. 177, October, p. 51 (TP215).

Matlock, L. H., and Dawson, R. F.
Aids in the Interpretation of the Consolidation Test—Summary of Proceedings of *STP 126*, 1249.

Mayne, C. R.
See Muhlenbruch, C. W., Krivobok, V. N., and Mayne, C. R.

Meloche, V. M.
A Review of Flame Photometry—Summary of Proceedings of *STP 116*, 1296.

Mielenz, R. C.
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Mielenz, Richard C., and King, Myrle E.
Identification of Clay Minerals by Staining Tests, 1213.

Mikhalapov, G. S.
Direct Explosion Test to Evaluate Load-Carrying Capacity of Welded Steel Structures. Published in ASTM BULLETIN, No. 172, February, p. 53 (TP67).

Miles, R. D.
Application of Aerial Photographs to Preliminary Engineering Soil Surveys—Summary of Proceedings of *STP 122*, 1250.

Miller, A. J.
See Armstrong, T. N., and Miller, A. J.

Miller, O. O.
See Seens, W. B., Jensen, W. L., and Miller, O. O

Mitchell, R. B.
Practical Evaluation of Soiled Test Pieces. Published in *STP 115*.

Moberg, M. L., Waithman, V. B., Ellis, W. H., and DuBois, H. D.
Determination of Calcium in Lubricating Oil by Flame Spectrophotometer—Summary of Proceedings of *STP 116*, 1296.

Mohr, C. F.
Discussion, 1137.

Moore, R. W.
Earth Resistivity Tests Applied to Subsurface Reconnaissance Surveys—Summary of Proceedings of *STP 122*, 1250.

Morgan, O. M.
Canvas Disk Method. Published in *STP 115*.

Morrison, H. L.
See Finley, G. S., Morrison, H. L., and Ragan, H. S.

Morton, P. H.
Discussion, 1059.

Mosborg, Robert J.
Apparatus for Low-Temperature Tension Tests of Metals. Published in ASTM BULLETIN, No. 177, October, p. 41 (TP205).
See Newmark, N. M., Mosborg, R. J., Munse, W. H., and Elling, R. E.

Muhlenbruch, C. W., Krivobok, V. N., and Mayne, C. R.
Mechanical Properties in Torsion and Poisson's Ratio for Certain Stainless Steel Alloys, 832. Disc., 854, 855.

Munse, W. H.
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See Newmark, N. M., Mosborg, R. J., Munse, W. H., and Elling, R. E.

Munse, W. H., and Weil, N. A.
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LIST OF SPECIAL TECHNICAL PUBLICATIONS

The publications listed below have been printed during the year 1951 and issued as separate publications not included in either Proceedings or ASTM BULLETIN. For a list of earlier Special Technical Publications, see the original listing in *Proceedings*, Volume 48, page 1352 (1948), and the supplementary listings in Volume 49, page 1240 (1949), and Volume 50, page 1490 (1950). A complete list of Special Technical Publications covering all publications, up to and including 1950, is included in the 50 year Index to ASTM Publications.

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60-A	Paper and Paperboard—Characteristics, Nomenclature, and Significance of Tests (1951).
114	Symposium on Bulk Sampling (1951.)
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116	Symposium on Flame Photometry (1951).
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127	Changes in Characteristics of Portland Cement as Exhibited by Laboratory Tests over the Period 1904 to 1950—H. F. Gonnerman and William Lerch (1951).